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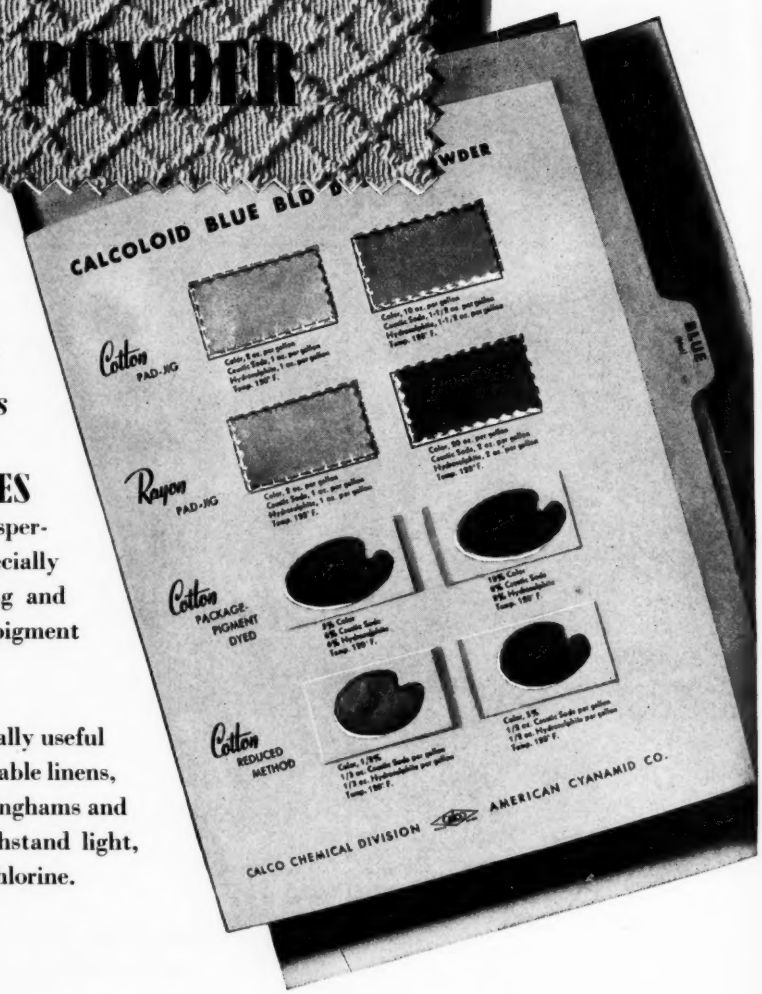

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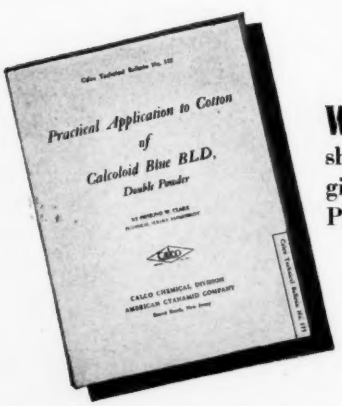
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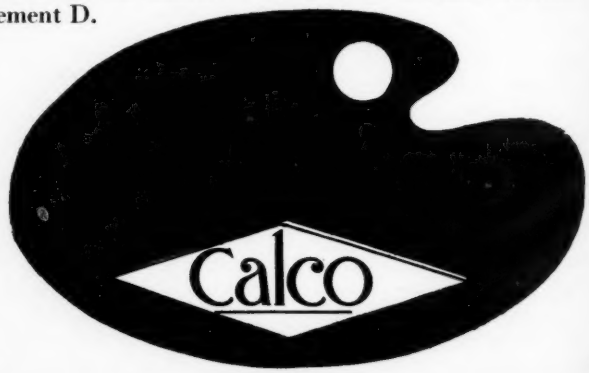

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VOLUME 30 MAY 12, 1941 NUMBER 10

The Effect of Wetting Agents on WOOL FELTING

JOSEPH W. CREELY and GEORGE C. LE COMPTE

THE idea of using surface active chemicals other than soap in felting is not new. Pinagel[9] patented the use of saponin for fulling in alkaline solutions in 1917. It wasn't until the early thirties that synthetic wetting agents came into general use. During this period Wagner[10] patented the use of alkyl naphthalene sulfonates together with metallic salts for working hair into felt. Böhme[1, 2] and also Johnson[5] patented the use of sulfuric acid esters of higher aliphatic alcohols in acid fulling liquors.

A study of the action of surface active chemicals on felting might have several uses. An obvious use is to make it possible to select materials which could act as aids for use in manufacturing wool felts. On the other hand wetting agents having a minimum of felting tendency are most suitable for use in a carbonizing bath. Using the latter a more open wool or noil is produced and more thorough dusting is accomplished after carbonizing. This study was made with the carbonizing problem in mind but the results might also have application in acid fulling.

EXPERIMENTAL PROCEDURE

Evaluation of Wetting Agents—The method employed here was the one suggested by the authors[3] for the evaluation of wetting agents for use in a carbonizing bath. The method consists of squeezing 2.0 gram samples of a uniform batch of noils in the solution to be tested. After the wool is wet out it is run through squeeze rolls and carefully placed on the surface of the carbonizing liquor held in a 600 ml. beaker. The complete sinking of the sample of noils is timed with a stop watch. The average of at least four such tests is used for purposes of comparison. There must be no foam on the surface of the liquid before running the test. The temperature of the liquid should be held constant in each case. 20° C. is a convenient temperature to use.

If sinking time is plotted against concentration of wetting agent on log-log paper a straight line is obtained for each different wetting agent. This is illustrated in Figure 1. Each point plotted in this figure is the average of four similar determinations of sinking time. As in all the work included in this article known amounts of wetting agents

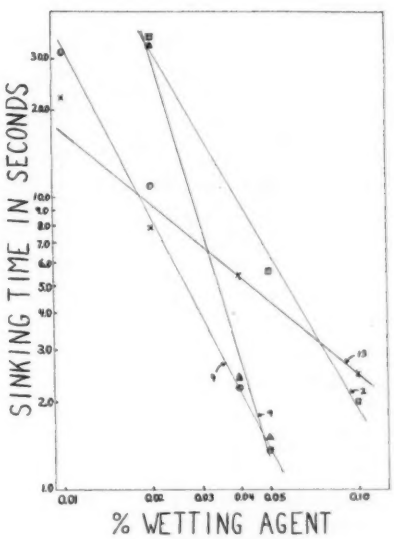

Fig. 1

in 4 per cent sulfuric acid was used because this approximates closely the strength of acid used in wool carbonizing.

Moisture in Wetting Agents—The method employed for determining moisture in the wetting agents tested was that recommended in the Yearbook of the A.A.T.C.C.[8]. Commercial wetting agents were studied and it is believed that water is the chief adulterant used. Sodium sulfate may be used in some cases.

Felting Studies—The method of evaluating the effect of the different acid baths containing wetting agents has been described in detail in a previous paper by the authors[7]. The method is a standardized shaking procedure in which measured lengths of yarn are shrunk in length and measured. By means of curves these measurements are used to determine the instantaneous rates of shrinkage of the yarns. A study of shrinkage-time curves showed that the instantaneous rates of shrinkage is constant over a large shrinkage range. The concentration of wetting agent used in the felting studies was such that a wetting time of 2.0 seconds was obtained in the test previously described. A fine two ply woolen yarn composed of ap-

proximately 70 per cent territory, 10 per cent pulled and 20 per cent Texas wool was used. It was purified by extraction with petroleum ether and was backwashed with dilute aqueous ammonia.

Briefly described the method is as follows: Samples of the wool yarn are cut into lengths of approximately one meter and loops tied on each end. Three such pieces are placed in a three ounce cylindrical wide mouthed bottle containing 50 mls. of the fulling bath. In this case 1 per cent sulfuric acid containing wetting agent was used. The yarn is allowed to soak 20 minutes to allow internal stresses to be relieved before measuring its length. The length of the yarn is conveniently measured by hanging it from a small nail even with the top of a vertically supported meter stick. A small hook weighing three grams is placed on the bottom loop of the yarn and the total length of the yarn read off the meter stick. Six lengths of yarn are taken for each test and their average length called L_0. The yarn is removed and folded double four times, then placed in the bottle and stoppered. The bottle is secured to a vane of an electric washer. For this work a temperature of 20° C. was used and it was unnecessary to add water and adjust its temperature in the washer. The washer is started and the bottle shaken for 10 minutes. The yarn is removed and carefully untangled and again measured. Call the average of the six such lengths L_{10}. The process is repeated for another 10 minutes on the yarn samples and the average of the six lengths after again measuring is called L_{20}.

A vane of the washer used in this work oscillated through a third of a revolution. Its period was slightly more than one second. The radius of the arc traversed by a bottle was 12.5 cms. The machine used had three vanes and could accommodate six bottles, hence 18 separate lengths of yarn could be used at one shaking.

S_{10} and S_{20} were defined by the following equations

$$S_{10} = \frac{L_0 - L_{10}}{L_0}, \qquad S_{20} = \frac{L_{10} - L_{20}}{L_0} \qquad (1)$$

We showed that the instantaneous rate of shrinkage $K_s = \frac{1}{L}\frac{dL}{dt}$ was constant* and could be calculated from S_{10} and S_{20} by means of the following equation

$$K_s = 0.0576 \quad [2\log(1\text{-}S_{10}) + \log(1\text{-}S'_{20})] \qquad (2)$$

where $S'_{20} = S_{10} + S_{20}$. This equation is cumbersome to use so we prefer to use a plot of K_s against S'_{20}. This is plotted in Figure 2 using several different ratios of S_{10} to S_{20}. Using this graph it is possible to get fairly accurate values of K_s from the data with very little work.

RESULTS

In Table 1 are listed the wetting agents selected for this study, the composition of the active material and the percentage of water present. Wetting agents numbered 13, 14, 15 and 16 were suggested by Kritchevsky's patents[6] on compounds of this type. The compounds were prepared by condensing the mixtures at 180°-190° C. for 20 minutes and cooling. The resulting products may have quaternary ammonium radicals in the molecule and are good wetting agents in an acid medium.

In Table 2 we compare the concentration of the wetting

*L is the instantaneous length of yarn and t is the time of felting.

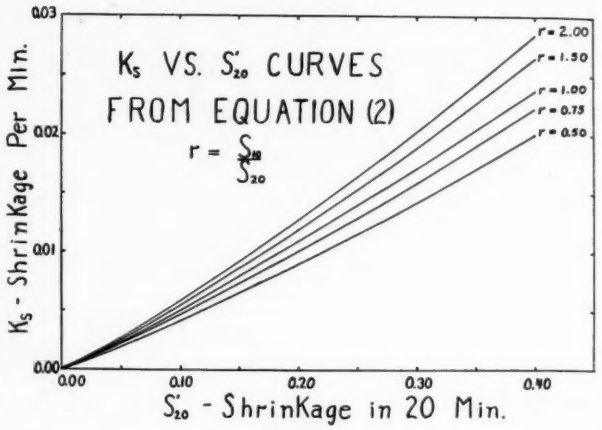

Fig. 2

TABLE 1

Wetting Agent	*Composition of Active Material*	*Per Cent Moisture*
1	Sodium salt of a sulfated fatty alcohol	70.0
2	Sodium salt of a sulfated branched chain aliphatic alcohol	60.0
3	$C_{17}H_{33}CONHC_2H_4SO_3Na$	80.0
4	Oleic acid ester of an aliphatic compound	5.0
5	Sodium salt of the sulfonated butyl esters of tea seed oil fatty acids	35.0
6	Sodium sulfonate of an ester of an unsaturated dicarboxylic acid	1.0
7	Sodium sulfonate of a C_{16} petroleum cut	1.0
8	Sodium alkyl napthalene sulfonate	7.0
9	Fatty acid amine	78.0
10	Castor amine	3.5
11	Coconut amine	5.0
12	Condensation product of 1 mole triethanolamine 1 mole oleic acid	0.0*
13	2 moles triisopropanolamine 1 mole oleic acid	29.5*
14	2 moles triethylene tetramine 1 mole oleic acid	27.2*
15	2 moles triethylene tetramine 1 mole coconut oil fatty acid	30.8*

*In these cases no moisture was present. The figures refer to the amount of excess amine present.

TABLE 2

Wetting Agent	*% Conc. Needed for 2" Sinking Time*	*% Anhydrous Conc. c*	S_{10}	S_{20}	K_S	*S.I.* x10⁴
1	0.02	0.006	0.121	0.082	0.0121	8000
2	0.10	0.04	0.076	0.084	0.0083	250
3	0.04	0.008	0.117	0.112	0.0130	7100
4	0.04	0.038	0.121	0.115	0.0132	1550
5	0.04	0.026	0.155	0.130	0.0172	3800
6	0.02	0.0198	0.141	0.092	0.0146	3680
7	0.05	0.0495	0.075	0.065	0.0076	61
8	0.02	0.0186	0.123	0.069	0.0120	2520
9	0.04	0.0088	0.082	0.074	0.0084	1250
10	0.02	0.0193	0.088	0.089	0.0096	1190
11	0.02	0.0190	0.114	0.097	0.0121	2500
12	0.01	0.01	0.087	0.050	0.0081	800
13	0.17	0.12	0.081	0.084	0.0088	125
14	0.05	0.0364	0.116	0.087	0.0119	1260
15	0.05	0.0346	0.110	0.088	0.0115	1210
Water			0.036	0.049	0.0040	
4% H_2SO_4			0.068	0.068	0.0073	

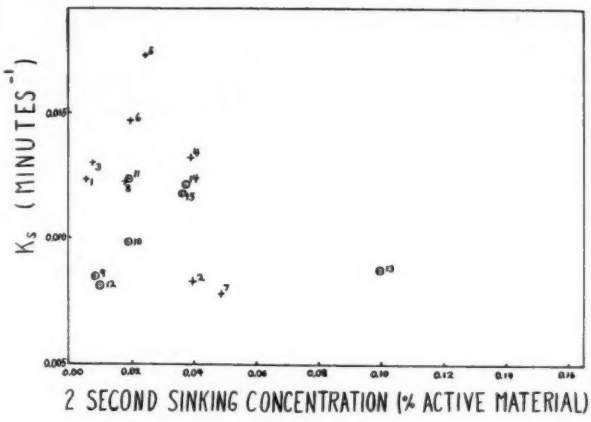

Fig. 3

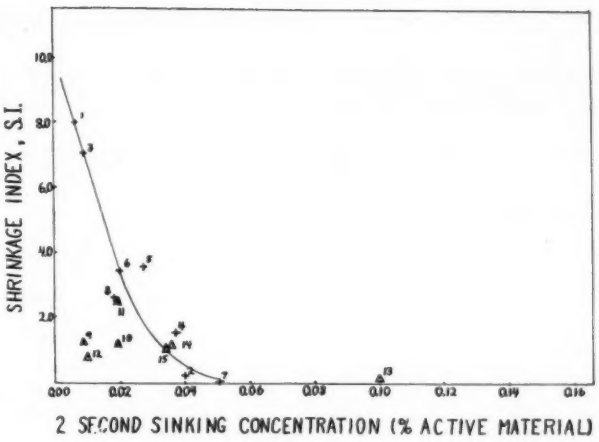

Fig. 4

agent to give a wetting time of 2 seconds, the shrinkage in the first 10 minutes shaking, S_{10}; the shrinkage in the second 10 minutes shaking, S_{20}; the instantaneous rate of shrinkage, K_s; the concentration of anhydrous wetting agent to give a wetting time of 2 seconds, c; and another quantity designated by S.I. We define S.I. for any material as being equal to K_s for that given material less the K_s found in a bath containing the same concentration of sulfuric acid but with no felting or wetting agent present, divided by the concentration of anhydrous felting or wetting agent used in the test.

$$\text{S.I.} = \frac{K_s - KH_2SO_4}{c}$$

It is to be noted that c and S.I. are quantities furnishing direct measures of the wetting and felting characteristics of the material. Thus, c is the concentration of anhydrous material required to accomplish a definite amount of wetting work in a definite time. S.I. is the amount of felting action brought about per unit concentration of anhydrous material.

In Figure 3 are found the plots of the various c and K_s values listed in Table 2. It will be noted that compounds 9 and 12 show 2 second sinking times at low concentrations and also exhibit little felting effect at these concentrations. These compounds therefore appear to be the most suitable wetting agents for use in the carbonizing of wool. Compound 9 is a fatty acid amine and compound 12 is the condensation product of 1 mole triethanolamine, 1 mole of oleic acid. It is interesting to observe that each of these materials is a cation active wetting agent. It is true that Figure 2 shows compounds 1 and 3 to be more effective in wetting than compounds 9 and 12, but compounds 1 and 3 also show a considerably greater tendency to increase the felting of wool and hence do not appear to be as desirable for use in carbonizing solutions.

In Figure 4 are found the plots of the various c and S.I. values listed in Table 2. It will be noted that compounds 1 and 3 exhibit by far the highest felting effects per unit concentration of material and therefore appear to be the most promising compounds for use as felting aids in acid solution. Compound 1 is the sodium salt of a sulfated fatty alcohol and compound 3 is $C_{17}H_{33}CONHC_2H_4SO_3Na$. Both materials are anion-active.

It will be observed that there is a very distinct negative slope to the curve drawn through the points of the anion-active materials plotted in Figure 3. It appears that if the wetting and felting tests described in this paper are applied to anion-active wetting agents, then good wetting agents will be found to be good felting agents and poor wetting agents will be found to be poor felting agents. These relationships are not nearly so definite in the case of the cation-active wetting agents. It is interesting to speculate upon the reasons for these relations between the wetting and felting characteristics of materials. On considering the wetting and felting tests used in this paper, it appears probable that the more highly adsorbed materials would tend to be better in both wetting and felting. High adsorptions should lead to subsequent short sinking times. High adsorptions should also lead to the formation of adsorbed films on the wool fiber which would be possessed of lubricating power and would therefore tend to yield more rapid rates of felting. With low adsorptions we should expect little effect on either wetting or felting.

Apparently then, it appears that the demonstrated relations between the wetting and felting effects of anion-active wetting agents (as measured by the methods described in this paper), are reasonable and to be expected. That these relations did not hold true for cationic-active wetting agents might be bound up with the fact that cations are not adsorbed by wool from acid solutions of pH less than 2.0[4].

CONCLUSIONS

1. It has been shown that wetting agents for use in wool carbonizing may have equal wetting powers and yet vary greatly in their tendency to make wool felt. Other things being equal, the wetting agent having least tendency to make the wool felt is most desirable.

(Concluded on page 268)

Relative Affinities of the Anions of

STRONG ACIDS FOR WOOL PROTEIN*

JACINTO STEINHARDT, CHARLES H. FUGITT, and MILTON HARRIS**

(Continued from issue of April 28, 1941)

2. Calculation of the Affinity of Anions for Wool

Any simple analysis of the experimental results in terms of the hypothesis of stoichiometric anion association depends on the assumption that the curve of acid combination is homogeneous, that is, the curve is determined by the combination of hydrogen ions and anions by sets of essentially similar groups in the fiber. If this is not the case, it is meaningless to ascribe shifts in the positions of the titration curves as a whole to variations in a single anion dissociation constant presumed to characterize each protein-anion combination. The assumption is empirically justified when the displacement in the positions of the curves affects all parts of the curve to the same extent. With the minor exceptions noted elsewhere the data in Figure 1 appear to justify a treatment based upon such homogeneity.

a. Relation of the Positions of the Titration Curves to the Anion Affinity

Since it has been assumed that differences in the positions of the curves with respect to the pH axis are determined by differences in K_A', the protein-anion dissociation constant, it is appropriate to attempt to evaluate K_A' for the anions of each of the acids used by means of the position of the corresponding titration curve. The reciprocal of K_A' is referred to hereafter as the anion affinity constant, or simply as the anion affinity.

It has already been shown[36] that changes in the positions of the titration curves obtained with a single acid which are produced by adding different amounts of the anion of the acid in the form of neutral salts can be described by the equation:

Fraction of wool combined =

$$\frac{[WHA]+[WH^+]}{[WHA]+[WH^+]+[WA^-]+[W^\pm]} = \frac{1}{1+\frac{K_H'}{a_H}\left(\frac{a_A+K_A'}{a_A+K_A}\right)} \qquad (1)$$

in which a_H and a_A represent the activities of the hydrogen-ion and of the anion respectively, the terms containing W represent ionic states of wool, and the constants K_H', K_A', and K_A govern the following hypothetical dissociation equilibria:

$$K_H' \quad WHA \rightleftarrows WA^- + H^+ \qquad (2)$$
$$K_A' \quad WH^- \rightleftarrows W^\pm + A^- \qquad (3)$$
$$K_A \quad WHA \rightleftarrows WH^+ + A^- \qquad (4)$$

The constant K_H governing a fourth possible equilibrium:

$$WH^+ \rightleftarrows W^\pm + H^+ \qquad (5)$$

does not appear in eq. 1, since it is obvious that the four constants are interrelated by the relation $K_H' K_A' = K_H K_A$.

The assumptions entering into the definition set down in the left hand part of eq. 1 have already been discussed[36]. It has also been shown that each experimental curve is actually somewhat broader (covering a wider range of pH values) than the S-shaped curves described by eq. 1, but that the displacement between curves obtained at different values of a_A are accurately described by the equation.* The difference between the curves obtained with and without a constant concentration of anions present is also accurately described by converting eq. 1 into the equivalent form, valid when salt is absent:

Fraction combined =

$$\frac{[WHA]+[WH]}{[WHA]+[WH^+]+[WA^-]+[W^\pm]} = \frac{1}{1+\frac{K_H'}{a_H}\left[\frac{a_H+K_A'}{a_H+K_A}\right]} \qquad (6)$$

The derivation of equations (1) and (6) made clear that their validity is limited to conditions under which the term WH^+ is greater than the term WA^-. These conditions are realized, except when much salt is present, when K_A' is greater than K_H. The latter constant has not been evaluated, but it must be considerably larger than K_H', which has been assigned the value 6.3×10^{-5} at 0° C.[36] Thus eq. 6 cannot be used for anions having affinities which result in a displacement of the titration curve of more than 1 or 2 pH units to the right of the position of the curve obtained with hydrochloric acid.

It is possible to derive an equation, similar to eq. 6 which has the complementary condition for validity, i.e., $[WA^-] > [WH^+]$, or $K_A' < K_H$. This equation, ap-

*Part of the material in the present paper was presented at the meeting of the American Society of Biological Chemists at New Orleans, Louisiana, in March, 1940[37].

**Research Associates at the National Bureau of Standards, representing the Textile Foundation.

*Reasons have been given for attributing this broadening of the experimental curves to the high degree of polyvalency of the wool protein[36], which has the effect of introducing an empirical exponent on the hydrogen-ion term in the simple equation[23]. The simplified treatment also necessarily leaves out of account all effects due to the mutual interaction of dissociating groups in the molecule except for the effect implied broadly in the inequalities $K_H' < K_H$ and $K_A < K_A'$.

(Continued on page 263)

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CALENDAR OF COMING EVENTS

Meeting, Northern New England Section, Andover Country Club, Andover, Mass., May 16, 1941. Speaker: Dr. Charles Hock, Chemical Foundation, Inc.

Meeting, South Central Section, Farragut Hotel, Knoxville, Tenn., May 17, 1941.

Meeting, New York Section, Swiss Chalet, Rochelle Park, N. J., May 23, 1941. Speaker: J. O. Amstuz, Behr Manning Corp., Troy, N. Y., "Electrocoating Fabrics."

Meeting, Rhode Island Section, May 30, 1941.

Outing, Northern New England Section, Vesper Country Club, Lowell, Mass., June 6, 1941.

Outing, Mid-West Section, Lake Lawn, Delavan, Wis., June 14, 1941.

Outing, New York Section, North Jersey Country Club, Paterson, N. J., June 20, 1941.

Outing, Philadelphia Section, Whitemarsh Valley Country Club, June 20, 1941.

Outing, Rhode Island Section, Norwich Inn, Norwich, Conn., June 20, 1941.

Annual Meeting and Convention, Carolina Hotel, Pinehurst, N. C., Oct. 31, Nov. 1, 1941.

Recent Advances in WATER TREATMENT*

ESKEL NORDELL**

THE subjects which this paper very briefly covers are : (1) the Automatic Zeolite Water Softener; (2) Carbonaceous Zeolites; (3) the Spaulding Precipitator, and (4) Anion Removal by the De-Acidite Process.

THE AUTOMATIC ZEOLITE WATER SOFTENER

The automatic zeolite water softener was designed not to eliminate the operator but to lighten the operator's work by eliminating the necessity of his presence at whatever odd hours a softener unit required regeneration. Also, it was designed to repeat with unvarying precision the various operations required. It accomplishes this by means of a motor-driven multiport valve coupled with electrical controls, the functioning of which is as follows:—

Softening Run

The length of the softening run is governed by an electric contact head meter placed in the soft water line. When the predetermined amount of water has been delivered by the softener, this meter closes an electric switch which establishes a circuit and causes the motor to rotate the multiport valve to the *wash* position.

Backwashing

This cuts the softener unit out of action and backwashes it. A rate of flow controller governs the rate of backwashing while the period of backwashing is governed by an electric time switch. At the end of this period the time switch closes, actuating the motor, which then rotates the multiport valves to the *brine* position.

Brining

In this position, a flow of water, passing through a hydraulic ejector, and acting on a pilot valve, injects the predetermined quantity of brine from the brine measuring tank into the top of the softener so that it passes downwardly through the softener and out to the drain, thus regenerating the zeolite by restoring it to its original active or sodium state. When this predetermined quantity of brine has been injected into the softener, a float switch in the brine measuring tank closes, thus establishing a circuit which operates the motor so that it rotates the multiport valve to the *rinse* position, during which the pilot valve automatically closes.

Rinsing and Returning to Service

In this position the softener is rinsed free of waste products and excess salt by a downward flow of water. The rate of rinsing is governed by a rate of flow controller, while the period of rinsing is governed by an electric time switch. At the end of the rinse operation this time switch closes, establishing a circuit which actuates the motor so that it rotates the multiport valve to the *soften* position, whence it is again automatically placed under the meter control, which governs the length of the softening run and initiates the next cycle of operations.

CARBONACEOUS ZEOLITES

A recent development in the zeolite process has been the production of non-siliceous zeolites, which may be used either on the sodium cycle or the hydrogen cycle. On the sodium cycle, these zeolites are regenerated either with salt or sea-water. On the hydrogen cycle, they are regenerated with sulfuric acid.

Zeo-Karb is typical of these zeolites. It is a sulfonated organic material, and may be used not only with normally alkaline waters but also with waters of low pH value. Since it is non-siliceous in nature, it eliminates the possibility of silica pickup from the zeolite bed.

On the sodium cycle, it behaves much the same as the older types of zeolite; that is the hardness salts and iron, and/or manganese, are replaced by the corresponding sodium salts. Similarly, also, it is regenerated with salt.

It is on the hydrogen cycle, however, that its behavior is quite different. For on the hydrogen cycle it removes the bicarbonates from water completely, leaving nothing in the water but carbon dioxide, which is then easily removed by aeration.

Further, it removes not only the bases calcium and magnesium but also sodium. Therefore it is possible to remove sodium bicarbonate from water without having to resort to distillation. Iron, and/or manganese, if present in soluble form, may be removed by Zeo-Karb on the hydrogen cycle in the same manner as with other zeolites on the sodium cycle.

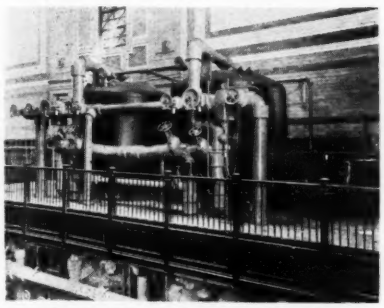
When the base-exchanging capacity of Zeo-Karb is exhausted, it is regenerated with a dilute solution of sulfuric acid. This removes the bases from the zeolite in the form of their sulfates and restores the hydrogen to the zeolite.

When water containing sulfates or chlorides in addition to the bicarbonates are softened through the hydrogen cycle, the corresponding mineral acids are formed. Therefore, in handling such waters, one portion of the Zeo-Karb plant may be worked on the hydrogen cycle and the other on the sodium cycle. The two effluents are then mixed in whatever proportions are necessary to yield a mixed effluent of the desired degree of alkalinity. If the chloride and sulfate content is low, it is often feasible to use only hydrogen zeolite and then neutralize with alkali.

THE SPAULDING PRECIPITATOR

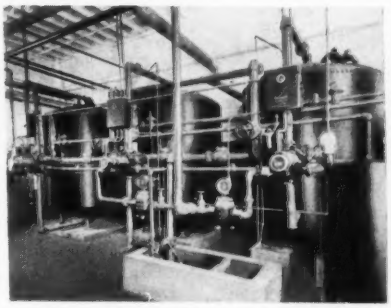
The Spaulding Precipitator represents the latest developments in cold lime soda water softening. In this, by

*Presented at meeting, South Central Section, Nov. 9, 1940.
**Technical Department, The Permutit Company.



Zeo-Karb Water Softener Installation

Spaulding Precipitator Water Softener Installation



Automatic Zeolite Water Softener Installation

means of bringing the treated water in contact with the suspended precipitates, through a novel design and operation of the settling tank, results are achieved which are impossible with the older types of lime soda water softeners. The size of the settling basin is reduced to a 1 hour detention period instead of the 4 to 8 hour detention period used in the older types; the lime is used more efficiently; the calcium bicarbonate alkalinity may be reduced to about 2 grains per gallon or less; stabilization may often be effected without recarbonation; and the size of the precipitates is so much greater that coarser sand, and consequently greater filtration rates, may be employed in the filters.

These results, as stated, are accomplished by utilization of the sludge from previously formed precipitates. In carrying out this process, the water, after being treated with lime or lime and soda, is then mixed with previously formed sludge by means of a mechanical agitator, situated in the bottom of the precipitator.

The mixture then rises through a sludge filter zone where it comes in intimate contact with a suspended sludge blanket. The sides of this zone are sloped so that the water, as it rises, spreads out and consequently its upward velocity decreases. It finally reaches a level such that the velocity is low enough to leave the sludge particles behind. Therefore the effluent emerges from the top of the precipitator clarified, softened and stable.

Due to the intimate contact of the sludge blanket with the treated water, stabilization of the water is effected as these conditions inhibit super-saturation. Also, due to their method of formation, the precipitated particles are coarser in nature and settle more readily. This zone of suspended precipitates functions likewise as an upflow filter, thus assuring a greater clarification of the treated water than could be obtained otherwise.

The mixing period in the Spaulding precipitator is 30 to 40 minutes. This is about three times as great as the older design. On the other hand, the settling period need only be form 20 to 30 minutes, which is only 1/8th to 1/12th of the settling period required in the older type. In the Spaulding design the sludge is removed through a simple concentrator chamber, thus eliminating the expensive sludge scraping or collecting systems formerly used. Also, due to the smaller size of settling tank employed, a Spaulding precipitator plant, including filters, occupies less than half the ground space formerly required.

Iron, Manganese, Color, Turbidity and Silica Removal

Iron and manganese, if present in soluble form, may be removed with the hardness in the Spaulding precipitator by aerating the water before it enters the precipitator. Iron and manganese, in suspended, organic or colloidal form, are often found in surface waters. These surface waters may be freed of these undesirable metals, and also color and turbidity, by coagulation and settling in the Spaulding precipitator.

In addition, the Spaulding precipitator has proven especially efficaceous in removing silica from water due to the intimate contact of the previously formed sludge with the water. In silica removal, various forms of dolomitic lime or other sources of magnesia may be employed as the silica adsorbing material.

ANION REMOVAL BY THE DE-ACIDITE PROCESS

De-Acidite is a zeolite which removes the acid radicles from water in much the same way as other zeolites remove the bases. By using De-Acidite in combination with Zeo-Karb on the hydrogen cycle, it is possible to produce an effluent which is practically a distilled water.

The method of operation is to use a Zeo-Karb water softener on the hydrogen cycle before the De-Acidite unit. The Zeo-Karb takes the sodium, calcium, and magnesium out of the water and leaves the corresponding radicles in the form of their respective acids in the water. Thus the bicarbonates leave the theoretical carbonic acid in the water where it promptly breaks down into carbon dioxide and water.

Similarly, the sulfates and chlorides form respectively sulfuric and hydrochloric acids. This acid effluent is then passed through the De-Acidite. The sulfuric and hydrochloric acids are taken up by the De-Acidite while the carbon dioxide passes through unchanged.

The effluent then is passed through a degasifier which removes the carbon dioxide thus furnishing an effluent which is practically a distilled water. The Zeo-Karb, when exhausted, is regenerated with sulfuric acid, while the De-Acidite, when exhausted, is regenerated with soda ash.

NEW METHODS OF PRINTING*

WILLARD L. MORGAN and NORMAN L. VAUGHN**

SHORTLY after the present war began, scarcities appeared in some of the printing dyestuffs and we will describe four new methods of printing which have been developed to take care of these situations and to further enhance the printer's art. Direct dyestuffs have been used in relatively small extent in printing for some time back. However, such prints suffered from low intensity due to relatively poor solubility of the dyestuffs and also to the tendency of such dyestuffs to readily wash out. Some time back the use of urea in a dye paste as a solvent for securing higher strength color in direct dyestuff printing paste was brought forth, and while this gave increased tinctorial power, the use of an aftertreating bath was also necessary in order to fix the dyestuff. The fixing agents ordinarily used, however, often alter the shade and almost invariably accelerate the fading by light.

Examination indicated that a new approach to the problem was necessary and research was started building new organic chemicals which would have the power of driving the dyestuff into the actual cotton filaments to a much deeper depth than had been secured before. It is found that when this is done just as also has recently been proved on acid dyes on wool with more uniform and deeper penetration into the fiber, wash resistance is markedly increased and crocking disappears. Between 50 and 60 entirely new organic molecules were synthesized, printing paste added, prints made, wash tests run and various other properties observed so that the desired properties could be secured to a maximum degree.

The typical printing paste formula involving the new chemical thus developed appears in Table 1. In addition to the new synthetic there is used urea, direct dyestuff normally around 2 to 3 ounces per gallon and on cottons a standard type of printing starch. The dry gum is cooked up to about 1¼ lbs. per gallon of paste. The dyestuff and urea are mixed together in a bucket and then wetted out in a small amount of water. The synthetic and remaining water are then added. The printing paste as applied to the machine for direct dyestuff printing is preferably somewhat thinner than that normally used for other dyes. After printing, the goods are run over cans to dry and then passed through a rapid ager for approximately five minutes steaming in order to set the dyestuff. Wet steaming for one hour or longer is then necessary giving better fixation the longer the time used. The cloth is then finished by passing it opened out through about six heavy spray washes of cold water thrown onto the surface, to remove surface colors and the gums, and the goods are then dried on cans. The spray wash is essential and important. The excess surface dyestuff and starch must be removed in order to secure good washability, as otherwise the whites will be redyed by later washings.

TABLE 1
Typical Direct Dye Paste for Cotton
To Make 1 Gallon of Paste

MIX A
2.5 oz. Direct Dyestuff
13 oz. Ahco DyPrint X
13 oz. Urea
2 lb. 3 oz. Water
MIX B
12 oz. dry Ahcogum RPG
4 lb. 5 oz. Water

In Table 2 there is given a range of dyestuffs suitable for application in this manner. The fading characteristic of the dyestuff is unaffected by the printing method and the dyestuffs listed all give good 40-hour fading tests. The prints made using the recommended dyestuffs will stand No. 2 wash. It is necessary to use the recommended dyestuffs if satisfactory wash resistance and 40-hour fading tests are to be secured. The penetration even on heavy fabric is very deep, as can be seen by the eight-color print exhibited. The wash test on this piece can be examined by those interested. It will be found that there has been no undesirable color movement. It will be seen from the sample which has been washed a second time that all of the slight color movement occurs in the first wash, indicating this to be small residual dyestuff which has not been completely washed away in the finishing. The prints will not stand No. 3 or alkali washes, but it is apparent that the process is applicable to many types of cotton goods and rayon where extreme resistance to washing is not necessary. The direct dye prints show no crocking and are resistant to dry cleaning. Due to the relatively small amount of dyestuff used per gallon of paste, the printing

TABLE 2
Recommended Direct Dyestuffs
Dyes Good to Light and Washing

Althouse Dyes	*Calco Chemical Co.*
Fastolite Yellow L	Calcodur Pink 2BL
Vegentine Fast Yellow R	Calcodur Scarlet 4BSY
Superlitefast Yellow NLC	Calcodur Blue SL
Fastolite Orange GL	Calcodur Orange GL
Vegentine Fast Brown S	Calcodur Orange EGL
Superlite Fast Blue GL	Calcomine Orange R Special
Superlite Fast Blue RL	Calcomine Sky Blue FFP
Azoanthrene Royal Blue L	*DuPont*
Azoanthrene Navy CW	Pontamine Fast Red 8BNL
Azoanthrene Navy BR	Pontamine Fast Blue SFL
Fastolite Grey BL	Pontamine Fast Blue 4GL
Superlitefast Grey GL	Pontamine Sky Blue 6B
Superlitefast Grey LVL	Pontamine Orange PG
Azoanthrene Scarlet O	Pontamine Fast Yellow RL
Azoanthrene Scarlet Y	Pontamine Fast Turquoise SGL
Superlitefast Red 3BL	Pontamine Fast Brown 4GL
General Dyestuff	Pontamine Fast Orange RLG
Fastusol Green LBB	Pontamine Fast Brown 2RL
Fastusol Orange L7CA	Pontamine Fast Orange EGL
Fastusol Yellow LRA	Pontamine Fast Grey BL
Benzo Fast Scarlet 4BSA	Pontamine Fast Grey 2GL
Brilliant Benzo Blue 6BA	

*Presented before Rhode Island Section, Jan. 31, 1941.
**Arnold, Hoffman & Co., Inc.

costs are attractive. As can be seen, the direct dyestuffs offer quite attractive colors.

In the next slide we show a typical paste for printing on rayon where a special thickener is used in order to secure the necessary soft handle. The prints are naturally brighter and are also more resistant to washing and fading.

TABLE 3
Typical Direct Dye Paste for Rayon
To Make 1 Gallon of Paste

MIX A	
2.0 oz.	Direct Dyestuff
13 oz.	Ahco DyPrint X
13 oz.	Urea
14 oz.	Water
MIX B	
52 oz.	Ahcogum RD
58 oz.	Water

As many mills do not have the cottage steamer or cannot arrange the long steaming time which the above method requires, a further modification has been worked out involving the use of the formaldehyde type setting direct dyestuffs. It is not possible to add formaldehyde directly to a solution of such a dyestuff without precipitating or resinifying the dyestuff to a condition where it will not penetrate into the fiber. Considerable investigation was necessary before a new chemical was found which could be directly added to the printing paste composition without harming the dyestuff or the stability of the paste in the presence of urea, and yet would allow deep penetration into the fiber of the dyestuff and thereafter resinifying and insolubilizing the same inside the fiber. In this method the printing paste was the same as that already indicated, excepting that the chemical, "Ahco DyPrint T," is used in place of the other synthetic. After printing, the cloth is run through an ager twice or for a total of ten minutes, and after that the goods are washed with six spray surface washes of cold water to remove unfixed dyestuff and printing paste as before.

Most of you are familiar with the use of pigments in printing and are well aware that compositions are available for use in connection with starch printing pastes which will set onto the goods by a baking operation, after which the starch is removed by washing. Such compositions are now available from several sources.

Pigments have also been printed onto cloth considerably by the use of water in oil dispersions. Extremely fine sharpness of mark has been claimed for this method, and the impression has been given that such sharpness can be secured only when such type emulsions are used.

It is our pleasure to introduce to this group an entirely new method of printing with pigments which will give very fine line, sharp prints without being a water in oil emulsion. In this method we take a clear, light yellow fluid which has the consistency of molasses, and merely stir various amounts of water into the same. The mix thickens as water is added, and any printing viscosity can be secured depending on the amount of water added. No cooking is necessary, and the pigments mixed with a little water if necessary are merely strained through a piece of cloth into the paste. The pigments are stirred into the paste and it is not necessary to use a homogenizer at any time.

Table 4 shows a composition for such a printing paste.

TABLE 4
Light Blue Pigment Print
To Make 1 Gallon of Paste

3¼ oz. Monastral Fast Blue BF
4 lbs. 4 oz. Ahcoprint F
Mix above and stir in
3 lbs. 10 oz. Water
Print and dry.

The pastes will not putrefy or sour on storage, and may be kept for long periods, as they do not dry out. The paste requires no special handling on the printing machine and after printing is merely dried on the cans with no baking and no ageing necessary. Also, no washing is necessary, as there is no starch or usual thickener to be removed.

The paste is very stable and not affected by machine running. As the paste is mainly a water proposition, the clean up on the roll is extremely simple and easy, the cleaning being done with water. No cleaning solvents are necessary, thus eliminating possibilities of dermatitis with the printers, and no stickiness is left on the rolls or other materials. The pastes are non-inflammable although solvents are present, the pastes have only slight effects on rubber and no effect on any of the metal parts of the machine.

The printed cloth shows very fine sharpness. It shows the full light resistance of the pigments used and it gives good crock tests. The printed cloth is finished with a soft handle and the wash test both to the standard No. 2 and No. 3 tests show practically no wash off of the pigment; that is, the pigment is tightly fixed onto the fiber. The printed piece which we are showing you has three colors; namely, red, dark blue and black in indigosols and rapidogens, and three other colors, namely; light blue, yellow, and green, all done using Ahcoprint F.

Many of the dyestuff pigments supplied by duPont, General Dyestuff, Calco and other firms such as those shown in Table 5 can be used.

TABLE 5
Pigments for Printing
Supplied as Pastes

DuPont	*Calco*
Monastral Fast Blue BF	Calcoloid Ptg. Yellow GC
Lithosol Fast Scarlet RD	Calcoloid Yellow 5GD
Lithosol Fast Brown V	Calcoloid Brown RRP
Lithosol Fast Yellow ARP	Calcoloid Grey RD
Lithosol Fast Brown R	Calcoloid Grey GD
Lithosol Fast Maroon 2BP	Calcoloid Black AD dbl.
Lithosol Yellow GP	Calcoloid Grey BD
Lithosol Blue GLR	Calcoloid Voilet 6RD
Lithosol Orange OTP	Calcoloid Violet 2RD
Lithosol Fast Orange 4C	Calcoloid Navy Blue NTD
Lithosol Acid Yellow 3G	Calcoloid Blue BLD
General Dyestuff Corp.	
Hanza Yellow GLD	Heliogen Green C
Lithol Fast Orange RD	Heliogen Blue BA

The materials used and the process of printing with them in this method are not water in oil type emulsions and are covered by U. S. Patents already issued and others applied for, and the methods and chemicals employed in the direct dye printing are also covered by patents applied for.

Methods of EVALUATING WOOL OILS*

CLARENCE BURTON KINNEY**

IT would be very interesting if we could step back to the early days of the factory production of woolen goods and the first application of oil to wool, particularly in water emulsion. At that time, the only oils obtainable in commercial quantities were saponifiable oils or oils containing some natural unsaponifiable matter. Mineral oils were unknown. The situation is entirely different at the present time. Not only do we have a vast variety of saponifiable oils produced in all parts of the world but we have mineral oils of unsaponifiable type and grade. We also have the so-called synthetic oils which are modifications of natural glycerides, fatty acids, and hydro-carbons. This condition has permitted an extensive opportunity to a compounder of oils, as evidenced by the number and variety offered to the trade. Even though the present war has either cut off supplies of certain oils or raised the cost to prohibitive figures, we still have a large variety of domestic products.

The woolen and worsted manufacturer is being offered stock lubricants of all types and claims. It is his task to pick out from these products the one or more best suited to his requirements. The factory evaluation of a stock lubricant is slow and expensive, so the manufacturer looks to his laboratory or chemist to guide him. The laboratory evaluation of a wool oil does not tell the entire story but can determine very necessary points. A mere chemical analysis does not by any means determine its suitability and in many cases is misleading and erroneous.

Practical tests, however, can be made which take little time and show characteristics which are essential and practical. It is not possible to go into detail regarding methods of evaluation, so I am only going to touch on the high spots.

Under date of December 17, 1937, Arthur D. Little, Inc., brought out a report, "Survey of Alternatives for Olive Oil in Process of Wool Manufacture," which is very complete and gives their methods of laboratory testing. A number of oils from different concerns were reported. As the object was a replacement for olive oil, naturally many of the oils, while not being suitable as substitutes for olive oil in every respect, might be very suitable for the textile industry where all the characteristics of olive oil were not required.

Anyone interested in testing methods of this nature can profit by reading this report. It deals with proprietary products, i.e., mixed oils prepared for the trade in the form of emulsifiable oils, also glycerides and blending mineral oils. Since that time many of the findings shown in the Arthur D. Little report could be greatly changed, but in spite of that, the methods of his valuation and the manner in which he went at the work are entirely correct and are a good example of accurate chemical analysis on a large scale.

In classifying wool or worsted oil, it is first essential to determine conditions under which the oil will be used in practice. It is true that all wool oils have to meet the usual tests in regard to lubrication, brittleness, fly, etc., but there are many other considerations which enter the picture. The man making shoddy does not require worsted oil, and a manufacturer of cheap woolen goods does not require the same oil as one making sweater yarn. A fancy piece goods manufacturer has different problems than the one making heavy grade of fulled fabric. Taking all of these conditions under consideration, we can lay down specifications which cover nearly all requirements and then pick out those desirable for each condition. We can enumerate in our list:

1. Whether it is to be a neat oil or emulsifiable oil.
2. Ability to emulsify.
3. Stability.
4. Scourability.
5. Odor.
6. Volatility.
7. Corrosive effect.
8. Lubricating ability.
9. Static properties.
10. Availability.
11. Price.
12. Stability of price.

These chief classifications can in many cases be subdivided, and we will take them in turn and briefly outline those tests that can be made without the necessity of plant application.

1. *Whether an oil should be a neat or emulsifiable oil* is determined by plant conditions. If it is desired that the oil be applied in both forms, as is the case in many instances, a neat oil that will emulsify readily with a mild alakli will meet both conditions. That particular test needs no laboratory evaluation or any work in the laboratory.

2. *Ability to Emulsify.* This test requires little comment, as a neat oil which can be emulsified easily with borax, ammonia, or other alkali meets that requirement. A self-emulsifying oil that is readily soluble in water and forms a stable emulsion meets this test.

The stability of emulsions is a subject about which much

*Presented before R. I. Section, January 31, 1941.
**E. F. Houghton Co.

has been written and is a subject for discussion. There is a general opinion that a self-emulsifying wool oil should make a ready emulsion in water which would be permanent for an indefinite period of time.

3. *Stability.* This test is one which includes several different phases: (a) First, we have stability from the standpoint of oxidation and yellowing. This test can be made in the Fade-Ometer with the ultra-violet lamp which gives the accelerated ageing test. Thirty hours in a Fade-Ometer is equivalent to six months normal ageing. If you do not have a Fade-Ometer, it is easy to rig up an apparatus by using the ultra-violet lamp which is common in many homes. Place a large wash tub under the lamp properly equipped with a reflector. Put ½" of water in the wash tub and the swatches to be tested around the sides of the tub. This produces results similar to those obtained from the Fade-Ometer. This test will definitely show whether an oil will oxidize on ageing.

(b) *Spontaneous Combustion.* This test is made by the Mackey test which must be done by someone equipped to do it. It is carried out by saturating cotton, wool or some other type of material with the oil to be tested. Placed in a closed container fitted with a thermometer, the temperature is raised from the boiling point and the rise in temperature recorded after this temperature has been reached. There are very few oils that will stand up under the Mackey test. However, the Mackey test is not the last word, for some oils will pass the Mackey test but will not be absolutely immune to spontaneous combustion. If it doesn't pass the Mackey test then its use is not recommended from the standpoint of spontaneous combustion.

(c) *Rancidity.* This test is made by saturating a fabric with oil to be tested. The sample is then placed in a closed bottle which is subjected to heat, usually a rise of temperature to 250° F., and then cooled down. The cap of the bottle is then removed and the bottle allowed to remain at room temperature overnight. This procedure is repeated for as long a test as desired. We have made tests over three weeks' time, each night and morning raising the temperature, removing the cap and putting it back on. It is not usually necessary to do it for that period of time because if an oil is going to be rancid it will usually do it within 24 hours under that heat. Sometimes rancidity will not show up in a week's time, but unless a person is ultra-conservative in regard to the question of rancidity, it is not necessary to continue the test for more than 48 hours.

If an oil will pass these three tests successfully, it is pretty sure that the oil is stable and will give no trouble in actual practice.

4. *Scourability.* Naturally, every manufacturer of woolen and worsted goods desires an oil that scours easily. Practically any oil can be removed if given drastic enough treatment, so the test for scourability should be one that will show both the difference in ease of removal and removal under the particular practical conditions under consideration.

The methods given by Arthur D. Little were conducted with yarn. We conduct our tests with scoured raw wool. As stated here, the question of scourability is one that is relative. In an ordinary shoddy mill they use so much soap and alkali to full the goods that almost anything will come out, but, nevertheless, an oil that would scour easy would not require that drastic treatment, so we make a test that will give the comparative ease of scourability.

We scour the yarn, or wool, in the Launderometer under standard conditions, usually 120° F., for a definite period of time, and then we make an ether-and-alcohol extract of the dried wool, or yarn. After scouring, we determine the residue and figure the percentage of oil removed under those conditions. This is as nearly a perfect evaluation as we have ever found.

We have set up another test where we use actual plant conditions. We make a soap and alkali solution of the same strength as used in the mill. We oil raw wool and then card it so that we get it absolutely uniform. We saturate this with the correct amount of soap-and-alkali solution and work it for a definite length of time, then wash it out and rinse it until free of soap. Then we determine whether or not that particular wool oil can be entirely scoured from the fabric. We then make an ether-and-alcohol extract and determine the residue and figure the percentage of oil removed under these conditions.

Another test made by Little is to dye a fabric, or spot the fabric first with oil, put it under the ultra-violet lamp, oxidize, scour and then dye with acid blue. Acid blue, which is Sulphon Blue, is very susceptible to the presence of oil and shows uneven dyeing, but this test is not entirely correct because you do not oil fabrics in that manner. You put the oil as uniformly as possible throughout the entire lot.

5. *Odor.* Regarding odor, this has been partially covered under the heading of "stability." However, many oils considered stable have an initial poor odor which is due to either natural odor or an added ingredient. The oil itself should have a satisfactory odor and not develop one on ageing, so this test is made by determining the odor at the outset, as well as the odor after ageing.

6. *Volatility* with neat oil and with emulsion. Water evaporates out of certain emulsions faster than others, and many manufacturers consider this important as they consider water a good lubricant. If they can retain a certain amount in the wool during the process they believe it improves their working conditions. We test in two manners.

First, by standard emulsion, three to one, or two to one, whatever is comparative with practical conditions, we permit this to stand at a uniform temperature, compare with a standard and determine rate of evaporation by decrease in volume.

Second, by taking neat oil itself and placing it in a

porcelain or some other type of dish not affected by heat. Permit it to remain at a standard temperature for a definite number of hours, starting at 10, 20, etc., and determine the actual loss of neat oil itself. This test will interest those who are putting oil in goods in order to obtain a little more weight.

7. *Corrosive Effect.* Chemical test for pH and total acidity will give certain information that will show the tendency for corrosion, but an actual test using card clothing will more nearly definite its action in practice. We can, also, include action on leather aprons. These tests require more time and the chemical tests for pH are considered sufficient preceding a plant application. It is difficult to determine the action of an oil on a leather apron unless it is actually put into practice. The question of corrosion on card clothing is one which is also subject to a great deal of debate.

8. *Lubricating Ability.* Here we may include the ability to reduce fly, supply spinning tenacity and yarn strength, also, viscosity and cold test of the oil. In the laboratory the cold test or pour test is easily determined. Only under conditions where unusual temperatures are found is the pour test particularly important.

The question of the correct viscosity is debatable because in our experience we have had oils which made thin emulsions but supplied a high degree of lubrication, and vice versa. This is a question of the emulsifying agent in many cases. So, these tests depend largely on the idea of the man who is laying out the specification. The best test is the plant trial.

9. *Static.* Here again, a plant test is the only satisfactory answer. It is not only in the woolen industry, but also in the rayon industry that this difficulty is found. In our experience, which includes the entire textile field, we have had a great deal of experience in the lubrication of rayon and there the question of static is very important.

10, 11, 12. *Price, Stability of Price, and Availability.* We cannot deal with these in the laboratory, but these points are readily ascertained from reputable sources of supply.

In evaluating a wool oil for any particular plant it is only necessary to scan these twelve different properties and pick out the important ones, then list them in the order of importance to you, to select the oil which best passes all the tests.

When the question of cost is of chief importance we definitely know that the cheapest oils will not pass several of the tests. When performance and not price is of first importance we can expect to get an oil which will pass all the tests. Logically, therefore, we should pick the exact requirements for the job and select the oil that will best meet the conditions.

MEMBERSHIP APPLICATIONS

Senior

Charles W. Dorn—Director of Research and Testing Laboratories, J. C. Penney Co., New York, N. Y. Sponsors: R. S. Lunt, L. A. Olney.

L. J. Todd Morris—Dyer, Horner Woolen Mills Co., Eaton Rapids, Mich. Sponsors: D. A. Anderson, B. H. Sharman.

Elizabeth D. Roseberry—Asst. Professor of Clothing and Textiles, Purdue University, West Lafayette, Indiana. Sponsors: C. R. Phillips, P. B. Mack.

Kenneth A. Sutherland—Colorist, European Textile Printing Co., New York, N. Y. Sponsors: Isaac Lovit, C. W. Kuhl.

Associate

William L. Baumner, Jr.—Salesman, Pioneer Salt Co., Philadelphia, Pa. Sponsors: E. C. Bertolet, J. E. Goodavage.

Howard M. Hall—Salesman, Marble-Nye Co., Worcester, Mass.

Student

John H. Hardwick—Philadelphia Textile School. Sponsor: E. C. Bertolet.

MEMBERSHIP, MAY 1, 1941

	Senior	*Junior*	*Associate*	*Student*	1941 *Total*	1940 *Total*	1939 *Total*	1938 *Total*	1937 *Total*
Northern New England	299	48	44		391	349	327	311	303
Rhode Island	255	38	27	17	337	332	295	284	256
New York	472	65	69		606	538	483	451	409
Philadelphia	379	49	47		475	447	381	369	326
Piedmont	249	25	23		297	293	277	247	238
South Central	61	5	5		71	61	63	56	41
Southeastern	95	14	29	4	142	137	133	109	55
Mid-West	147	20	25	1	193	180	157	158	147
Lowell Textile Institute				43	43	31	25	19	11
North Carolina State College				17	17	15	8	10	5
Philadelphia Textile School				20	20	13	18	28	25
No Section	66	9	12		87	80	71	65	55
	2,023	273	281	102	2,679	2,476	2,238	2,107	1,871
Corporate members					33	33	32	30	30
				Totals.	2,712	2,509	2,270	2,137	1,901

MEETING, PHILADELPHIA SECTION

THE last meeting of the Philadelphia Section was held at the Penn Athletic Club on Friday, March 14th.

We had as our speaker Philip H. Stott of the Du Pont Company, whose subject was "The Dyeing of Nylon."

The attendance for dinner was 131, while 146 members attended the meeting following the dinner.

Respectfully submitted,

J. P. CONAWAY, *Secretary.*

TRADE NOTES • NEW PRODUCTS

• GENERAL RELEASES

General Dyestuff Corp., 435 Hudson Street, New York, N. Y., announces release of circulars describing the following products:—

Indanthren Printing Black BBA Suprafix Conc.—a highly concentrated vat printing black, said to represent a grit-free, non-drying, non-settling paste. This product is offered in response to the constantly increasing use of shallow photoengravings on printing rollers. It is said to facilitate the production of deep shades of black under these conditions. Circular G-243.

Benzoform Orange FGL—a direct color recommended by the manufacturers for the dyeing of cotton and, in particular, of rayon. When aftertreated with formaldehyde and acetic acid, in the way usual for Benzoforms, it produces bright shades of orange said to be of comparatively good fastness to washing, to water, and to perspiration. The product is said to be well suited for use on materials which require a degree of fastness to wet processing beyond that obtainable with the average direct color. It is not recommended for discharge work. In union dyeings at the boil, the wool is dyed yellower and considerably weaker than the vegetable fibers, while at 150° F., it is only slightly stained and can be kept white by the addition of Katanol WB Conc. to the bath. Silk effects are dyed somewhat yellower than the vegetable fibers; acetate rayon effects are but very slightly stained. The addition of chrome to the formaldehyde bath affects neither shade nor fastness of the dyeings; copper sulfate renders the shade yellower and duller and affects somewhat unfavorably the fastness to light. Circular G-245.

Rapidogen Scarlet FFG — produces bright, yellowish shades of scarlet said to be of good fastness to chlorine and of very good fastness to washing and light. Like the other "FF" brands, it is said to show a high speed of coupling, being completely developed by a short acid ageing. Circular G-247.

Phenamine Black BD Extra—a direct dyestuff said to be well suited for the production of greenish blacks on cotton, rayon and other vegetable fibers. It is said to possess the advantage of slightly better dischargeability than the ordinary direct deep blacks. It can also be used for the dyeing of dischargeable blacks on both pure and tin weighted silk. Circular G-248.

Diazo Brown 3RNA—produces, when diazotized and developed with Developer AMS or Developer MT, reddish shades of brown said to possess fairly good fastness to washing. It is suitable for dyeing the various forms of cotton or rayon. It can be discharged to a fairly good white with Rongalite C. Circular G-249.

Diazo Fast Yellow 3GA—produces, when diazotized and developed with Developer ZA, bright, greenish shades of yellow said to be of fairly good fastness to washing and light. It is suitable for dyeing the various forms of cotton or rayon. It can be discharged to a very good white with Rongalite C. Circular G-250.

Benzoform Blue BR Extra—a direct color recommended by the manufacturers for the dyeing of cotton and, in particular, of rayon. When aftertreated with formaldehyde and acetic acid, in the usual manner for Benzoforms, it produces bright shades of reddish blue said to be of comparatively good fastness to washing, water and perspiration. The product is said to be well suited for use on materials which require a degree of fastness to wet processing beyond that obtainable with the average direct color. It is dischargeable to a very good white with Rongalite C in either neutral or alkaline pastes. In union dyeing at the boil, the wool is dyed somewhat weaker and redder than the vegetable fiber, while at a temperature of about 150° F. the wool is but slightly stained and can be kept practically white by the addition of Katanol W or WB Conc. The addition of chrome to the formaldedehyde bath affects neither the shade nor the fastness properties; copper sulfate turns the shade somewhat redder and improves both fastness to light and fastness to washing. Circular G-251.

• QUARTERLY PRICE LIST

The *Quarterly Price List*, publication of the R & H Chemicals Dept., E. I. du Pont de Nemours & Co., Inc., for April, 1941, has recently been distributed. Copies are available upon request.

• MEETING, S.O.C.M.A.

The Synthetic Organic Chemical Manufacturers Association of the U. S. held its regular monthly luncheon meeting at the Chemists' Club, New York, N. Y., on April 15th, 1941. The secretary, C. A. Mace, reported on various matters of current interest to the association including customs cases, patents, reorganization of the Bureau of Foreign and Domestic Commerce and monograph number 18 of the Temporary National Economic Committee which covers a "Trade Association Survey."

• CONSUMER TEXTILE TESTING COURSE

For the fourth summer now, the laboratories of the United States Testing Company, Inc., will be opened to a practical course in textile testing arranged by Miss Rajean Codish, in charge of the Consumer Service Division. It will give applicants an opportunity to put their textile knowledge to actual practice and learn, at first hand, the operation and theory behind many of the current textile testing devices.

Students will have personal instruction by experienced technicians in the company in classes which again are under the supervision of G. R. Turner, Supervisor of the Company's Textile Laboratory and Instructor of Textile Testing at Columbia University. The program of study will include theory and practice in demonstration and use of standard testing equipment. Analysis and testing of hosiery, fabrics and clothing, will be the basis of instruction. The testing of woven and knit fabrics for such factors as construction, thread count, fiber identification, tensile strength, seam slippage, color fastness to light, washing and drycleaning will be covered. A study of the new testing equipment recently developed, such as the Warmth Tester, Snag Tester, Torture Machine on Mattresses, and various shrinkage devices, will be an important part of the course. Students will be made acquainted with A.S.T.M., United States Government, Army, Navy and other standards.

This year's classes will be held from July 14th, through the 25th, excluding Saturday and Sunday, during the company's regular business hours from 9:00 A.M. to 4:00 P.M. There is no charge for the course, but students are expected to meet their own living expenses and transportation expenditures for the two-week period.

The group is limited in number to 20 students, in order that they may have the advantage of individual instruction and the opportunity to work with actual testing equipment. Applicants are required to have some elementary and textile training in order that some major portion of the two weeks may be devoted to an intensified study and use of testing apparatus.

Representatives of the fields of education, industry, and retailing are invited to take the course, applications for which may be obtained from the Hoboken office.

• SUMMER COURSE IN TEXTILE TESTING

A special summer course in Principles of Textile Analysis is again announced

by the Massachusetts Institute of Technology. The comprehensive program will be under the direction of Professor Edward R. Schwarz, in charge of the Textile Division, and will continue for six weeks beginning July 28th.

Laboratory exercises as well as lectures and demonstrations will cover the complete microscopic analysis of textile materials; physical testing, including heat transmission, permeability to air abrasion, flexibility, drape, water resistance, fastness to light and washing, resilience, interpretation of data, and application of statistical methods to textile testing. So far as possible the material will be presented in a manner to be of interest and value to the laboratory technician, to the manufacturer, to the teacher of this and allied subjects, and to the student who seeks a short orientation course in textile research and testing.

Opportunity will be afforded to study the new electron microscope, stroboscopic photography, stereoscopic photomicrography, and the spectrophotometric color analyzer. New equipment for refractive index measurement, single filament testing, resilience, heat transmission, permeability, photo-electric twist measurement for single yarns, fluorescent lighting for photomicrography, cotton maturity, wool quality, and rapid sectioning are also available.

Previous courses have been very successful and the present popularity of the work in textile technology is evidenced by the fact that the Engineering Defense Course in Textile Testing was oversubscribed by four to one and is now being repeated for a capacity group. Registration material for the summer session course and information concerning the work are available from the Summer Session Office, M. I. T., Cambridge, Massachusetts, upon request and applications to the extent of the laboratory capacity will be accepted in the order of receipt.

• TRANSFERRED TO RAYON DEPT.

Dr. Luther B. Arnold, Jr., has been transferred from the Technical Laboratory of the Organic Chemicals Department, E. I. du Pont de Nemours and Company, Inc., at Wilmington, Delaware, to the Technical Division of the Rayon Department, Richmond, Virginia.

• TEXTILE FOUNDATION DIRECTORS MEET

The 11th annual meeting of the Board of Directors of the Textile Foundation took place in Washington, D. C., on April 16th. Present were Franklin W. Hobbs, who was elected chairman of the Board for the 12th consecutive term; Frank D. Cheney, treasurer; the Under Secretaries of Agriculture and of Commerce; Edward T. Pickard, Secretary; and as consultants, Dr. Warren E. Emley of the National Bureau of Standards and Dr. R. Y. Winters of the Office of Experiment Stations, Department of Agriculture.

In addition to routine annual business the Director received progress reports on the work of the Foundation's research fellows at the National Bureau of Standards and at the Massachusetts Institute of Technology. At Standards, nine research authorizations are being worked on by 15 research fellows and at M. I. T., four fellows are giving attention to mechanical and optical properties of textile fibers. Published reports were received entitled "A One-Year Textile Course for College Graduates" and "What to do about Denim Stocks," the latter being the first in a series of seven reports under the general title of "Inventory Policies in the Textile Industries."

The Textile Foundation was organized for scientific and economic research for the benefit and development of the textile industry and its allied branches, including raw materials. The Directors of the Foundation are: Franklin W. Hobbs of Boston, chairman; Frank D. Cheney, Manchester, Conn., treasurer; Donald Comer, Birmingham, Ala.; the Secretary of Agriculture, Claude R. Wickard; the Secretary of Commerce, Jesse H. Jones; and Edward T. Pickard, secretary.

• DCAT ANNUAL FALL MEETING

At a meeting of the Executive Committee of the Drug, Chemical and Allied Trades Section of the New York Board of Trade held on April 24, the decision was made to hold the 6th Annual Fall Meeting and Golf Tournament at Skytop Club, Skytop, Pennsylvania, October 23-26, inclusive. For the past several years the Section has held very successful meetings at Skytop and the committee was unanimous in making its decision.

A program committee consisting of Victor E. Williams, Chairman, J. P. Remensnyder, S. B. Penick, Jr., and John C. Ostrom, Secretary of the Section, was appointed by the Chairman, John J. Toohy, of E. R. Squibb & Sons, to select an interesting panel of speakers.

The usual golf tournament and other interesting features which include a varied and entertaining program for the ladies are being scheduled.

• JOINS BURKART-SCHIER

Nelson A. Fisher, formerly overseer of dyeing at the Rock River Woolen Mills, Janesville, Wisconsin, has been named technical and sales representative of the Burkart-Schier Chemical Company for the mid-west area. Mr. Fisher has had many years of experience in the dyeing and finishing of textiles and should make a valuable addition to this concern.

The technical and sales organization of the Burkart-Schier Chemical Company is now composed of the following: C. A. Schier, W. A. Bentel, W. J. Kelly, Jr., Lawrence Newman, George McCarty, J. A. Brittain, T. A. Martin, H. V. Wells, George Rodgers, and Nelson A. Fisher. "Burk-Schier" Textile Chemicals include a complete line of wet processing agents. Plants are located at Chattanooga and Nashville, Tennessee.

• WORK OF CONSULTING CHEMIST

Karl M. Herstein, President of Herstein Laboratories, Inc., 18 East 41st Street, New York City, addressed the Student Affiliate Chapter of the American Chemical Society, at the Newark College of Engineering, on Friday evening, April 25th. The subject was "The Work of the Consulting Chemist."

• SPEAKS TO STUDENTS

Dr. Walter M. Scott of the Southern Regional Laboratory, New Orleans, visited the textile department of the Georgia School of Technology on April 23, and gave a most interesting talk to the junior and senior students in the department. Dr. Scott is director of the chemical finishing division of the new government laboratory, and his talk dealt primarily with some of the recent developments in the finishing of cotton fabrics.

Among the subjects discussed were methods for imparting a permanent glaze by using thermoplastic resins; making fabrics more crease resistant with synthetic resins; imparting a permanent stiffness or body with cellulose esters and ethers; water-, mildew-, and flame-proofing; and a number of others. Dr. Scott illustrated his talk by the use of numerous fabric samples, and at the close of the meeting, an informal session was held for questions and inspection of the samples.

• JOINS COMMERCIAL SOLVENTS

Commercial Solvents has announced that Ralph L. Ericsson has been added to the staff of their Trade Relations Division, Terre Haute, Indiana. His activities in his new position will be chiefly concerned with advertising and sales promotion. Mr. Ericsson is a graduate of Carnegie Institute of Technology and was formerly employed in the technical service division of the Warner Chemical Company.

• TECHNICAL NOTES FROM FOREIGN SOURCES •

War Time Printing Colors

M. Adanil—R.G.M.C.—520-154, *May*, 1940. — Although at the outbreak of the present war France was fortunately not faced with the same shortage of dyestuffs which had been experienced in 1914, the author, a veteran chemist-colorist, feels it worth while to give in detail a few printing formulas covering the application of certan well-known colors whose value has been established by long use.

In spite of the recent extensive employment of such colors as aniline, diphenyl, azoic and others in the production of blacks, logwood and its derivatives have remained of considerable importance for this purpose. Shaded with quercitron or Persian berry, they yield full tones of great beauty, depth and purity, are easy to apply in either roller or screen printing, and have no destructive effects on fabrics. Logwood extract as such is not usually employed in printing, as its color pastes soon form lakes and cannot be kept with safety for more than a few days. For this reason it is more generally applied as a reduced or carmine black, in which form it keeps well and as a steam color gives excellent results.

A disadvantage of the reduced logwood blacks, however, is the fact that they do not work well in company with the azoic printing compounds, as the bisulfite present in the black causes a rapid decomposition of the diazo if the two colors come into contact. If in printing the black carries over into the diazo there is immediate trouble, a gummy mass being formed which builds up on the roller and collects on the doctor blade. This difficulty may be surmounted by interposing a gum roller between the two colors, but it is no doubt safer in such circumstances to use a straight logwood extract containing no bisulfite.

For this purpose it is recommended to prepare a standard color paste with logwood and quercitron extracts, to a portion of which just before printing is added chlorate of soda and nitro-acetate of chrome in separately prepared solutions. Detailed instructions are given for making up this paste, which is said to be well suited for application to naphthol prepared fabrics, not to affect diazo compounds with which it is used, and with three or four minutes steaming in the Mather and Platt to give a black which is fast to boiling soap. It is noted, however, that the final printing color will not keep more than a day or two, so it should be mixed in the quantity required just before use.

If reduced logwood blacks when printing on white grounds give trouble with any of the other colors used because of the bisulfite present, it is possible here also to employ a pure logwood extract paste, though somewhat less chlorate and acid is recommended in the formula, and a more extended steaming required to compensate for the reduction.

The nitroso colors are encountered under various names and are of several types, such as the nitroso-naphthols, dinitroso-resorcin and dinitroso-dioxy-naphthols. Of these the dinitroso-resorcin is the most widely known and commonly used, as it deserves to be because of its cheapness, ease of application and excellent qualities of the colors it yields. It can be readily shaded with practically all of the alizarine colors and with several of the chrome colors as well. By combining it with different mordants such as iron and chrome useful shades of olive, brown and the like are readily formed.

Of the various diazo compounds, paranitraniline remains the most widely employed. To prepare stable diazo compounds of this and similar products the author prefers the use of ice. The base is dissolved in hot water and hydrochloric acid, and on cooling and adding ice is reprecipitated as a finely divided powder. Solid nitrite in fairly large pieces is then gradually added, the lumps sinking to the bottom and the liberated nitrous acid bubbling up through the entire body of the liquid, being thus completely utilized. If stored in a well covered container, the diazo compound thus prepared will keep almost indefinitely. Diazotized paranitro-orthoanisidine, benzidine, tolidine and dianisidine also keep well on storing; alpha-napthylamine, however, cannot be trusted for more than four or five days.

In applying such compounds it must be kept in mind that the stability of the diazo is ended when acetate of soda is added, and that they should be used immediately thereafter. The slight excess of acetate which is usually employed in printing pastes of this type is intended to afford protection to the back grays, which are otherwise rapidly attacked, though the print fabric itself is well guarded by the alkali present in the naphthol prepare.

Full instructions are given throughout for making up the various printing pastes and mordants which are discussed. The article was obviously prepared before the military collapse of France, and under present circumstances it is to be regretted that we cannot be certain what part of the French textile industry remains in condition to avail itself of the author's well meant suggestions.

Effect of Humidity on Yarns Made From Various Fibers

Ernst Thiemann, M. Textilberichte 21, 61, 1940.—This article contains a type of information sometimes wanted but difficult to find. The author has presented an unusual comparison of the effects of humidity on the strengths and elongation (at the break) of a number of yarns made from different materials. While such a comparison is necessarily not "accurate" the figures probably give a good picture of the relative values.

Kind of yarn	Relative Humidity						
	0	50	60	70	80	100 (wet)	
	Strength in grams		Change of strength				
Wool	237	6.7	10.1	14.8	19.3	29.5%	loss
Hair	3404	15	17.9	20.3	23.5	40.4	loss
Silk	912	10	12	13.8	16.8	35.4	loss
Cotton	604	4.3	9.4	15	21.5	25.6	gain
Flax	606	16.5	21.7	32.1	43.2	59.9	gain
Hemp	2988	37.9	47.7	59.9	69.4	87.6	gain
Jute	1573	17.4	30	45.7	40.3	4.1	gain
Ramie	1780	3.7	7.1	8.8	12.2	22.4	gain
Kapok	169	10	18.3	23	26.6	29	gain
Paper	1956	8	11	7.2 gain	0.4	41	loss
Vistra staple	158	2.5	4.4	8.2	15.8	43	loss
Copper rayon	182	11	16.4	20.8	27	48.3	loss
Viscose rayon	169	21.3	26	30.7	37.8	69.7	loss
Acetate rayon	145	9.6	13.1	17.9	25.5	40	loss

Kind of yarn	Relative Humidity						
	0	50	60	70	80	100	(wet)
	Extension in mm.		Gain in extension				
Wool	68	44.1	58.8	75	95.5	210.2%	gain
Hair	47	100	125.5	155.3	253.1	325.5	
Silk	42	16.6	19	23.8	28.5	50	
Cotton	32	3.1	6.2	9.4	15.6	40.6	
Flax	8	6.2	10	15	22.5	37.7	
Hemp	11	14.5	22.7	29	36.3	45.4	
Jute	7.5	2.6	4	12	16	1.3	
Ramie	13.1	16.0	14.5	19.8	25.6	53.4	
Kapok	22.6	22.9	41.5	56.6	70.3	88.9	
Paper	13.3	5.2	11.3	20.3	35.3	80.4	
Vistra staple	63	3.2	6.4	7.9	9.5	11.1	
Copper rayon	74	10.8	13.5	16.2	21.5	55.2	
Viscose rayon	78	23	28.2	34.5	43.5	93.5	
Acetate rayon	108	3.7	6.4	11.1	20.3	51.8	

The percentages of change due to different relative humidities have been calculated from the strengths and extensions observed when the yarns were tested dry. Owing to the great differences in the materials from which they are made the yarns themselves are, of course, not strictly comparative. The original has curves showing the changes in strengths and elongations and gives the observed values for all points for which we have here given only the percentage changes. Enough is reproduced here in a new tabular form for the reconstruction of any of the original data. To explain, under "Strength in grams" is given the breaking strength of the yarn when dry. The figures under "Change of strength" are all percentages of the "dry" values and represent gains or losses as the case may be. Then, for example, from the 237 grams breaking strength of wool and the 14.8 per cent loss at 70 per cent relative humidity the strength at this humidity is easily found. If it is a gain, as in the case of cotton, 15 per cent would be added to the 604 recorded, to obtain the strength at 70 per cent relative humidity. The elongations can be found in the same way.

The figures for strength in grams are apparently not strictly comparative. For example, the hemp yarn must have been much heavier than the flax. However, the *percentages* are presented as comparative. It is understood that the elongations as given in millimeters are comparative; at any rate the percentage changes in elongation are.

From the two tables given here the points of greatest change in either strength or elongation can be identified as well as the general course of the changes which take place. Estimates can be made of certain probable effects when one kind of yarn is substituted for another.

Jute shows the peculiar effect of an increase in strength up to 70 per cent relative humidity and a falling off at the higher values. Paper yarn increases a little in strength up to 60 per cent relative humidity and then goes off badly. The changes in extension were all gains except for wet jute. It would have been helpful if the elongations of the dry yarns had also been given as percentages of original length. The figures are not to be confused with stress-strain data; they are ultimate elongations; the 100 per cent for hair at 50 per cent relative humidity means that there is twice as much stretch and not that the sample became twice as long.

Improving the Durability of Spun and Filament Rayons

Quehl and Lampel, M. Textilberichte 21, 295, 1940.—Chemical companies and cloth finishers have done a great amount of work in the field of rayon, especially with spun yarns and fabrics, to impart to them the characteristic and valuable properties of wool and cotton. A great need is resistance to repeated washing, comparable with that of cotton, and resistance to abrasion. Up to the present time a treatment such as the addition of a resin, which has made an improvement in some particular direction has usually failed to develop other necessary properties or may have introduced new defects.

This article describes the results of a treatment where no foreign material is added but where a physical change in the cellulose of the fiber brings out a great increase in durability. It is the Adry process of the Theodore Rotta Company, Zwickau, Germany, and consists essentially in swelling the sensitive regenerated fiber in the yarn or piece by means of a controlled swelling agent which will not go too far; then washing the chemical out with water after it has served its purpose. The nature of the material used is not disclosed but the character of the results which are claimed might make it a desirable process if the use of it could be obtained here. It is probably not available at present and this brief abstract may prove suggestive for something else along a similar line.

The process may be operated on ordinary equipment and is simple enough to use anywhere. Its success depends upon the cellulose not returning to its original state, and upon its having better properties in its new form. The volume is greater and this gives a fuller hand which can be controlled to give stiffness if desired. The final dried cellulose seems to have a changed surface which suggests the cuticle of cotton or the outer layer of the

(Concluded on page 268)

(Continued from page 250)

plicable to acids with high affinity for wool, is obtained by equating the fraction combined to

$$\frac{[WHA] + [WA^-]}{[WHA] + [WA^-] + [WH^+] + [W^\pm]}$$

instead of to

$$\frac{[WHA]}{[WHA] + [WH^+] + [WA^-] + [W^\pm]}$$

and leads to the general result*:

$$\text{Fraction combined} = \frac{1}{1 + \frac{K_A}{a_A}\left(\frac{a_H + K_H}{a_H + K_H'}\right)} \qquad (7)$$

or, in the absence of salt, to the equivalent form:

$$\text{Fraction combined} = \frac{1}{1 + \frac{K_A}{a_H}\left(\frac{a_H + K_H}{a_H + K_H'}\right)} \qquad (8)$$

The dependence of the pH coordinate of the midpoint of each curve on K_A' or K_A, in terms of either eq. 6 or 8, may be described by equating the right hand member of each to 0.5. When this is done with eq. 6 the result is

$$K_A' = \frac{a_H(a_H + K_H)}{K_H'} - a_H = \frac{a_H^2 - a_H K_H'}{K_H'} \cdot \frac{K_H}{K_H - {}_H} \qquad (6')$$

With eq. 8 the result is

$$K_A' = \frac{K_H}{K_H'} \cdot a_H \frac{a_H + K_H'}{a_H + K_H} \qquad (8')$$

Eqs. 6′ and 8′ both contain constants which have not previously been evaluated (K_A and K_H) as well as the constant K_H' to which the values 6.3×10^{-5} has previously been assigned**. Because of electrostatic requirements, however, $K_H > K_H'$ and $K_A' > K_A$[36]. The ratios between pairs of dissociation constants which differ mainly because of the electrostatic effect of a single charge (such as the first and second dissociation constants of symmetrical dicarboxylic acids) always lie between 10 and 1000. If this analogy is accepted K_H probably lies between $10^{-3.2}$ and $10^{-1.2}$. Since the factor $\frac{K_H}{K_H - a_H}$ in eq. 6′ must be positive, the curves obtained with acids of lower affinity than hydrochloric acid (not reported in this paper) further restrict K_H to values greater than than $10^{-2.1}$. The factor thus approaches unity for values of a_H at the midpoints of practically all the curves represented in Fig. 1. Thus, between limits represented by the midpoint of the hydrochloric acid curve and the midpoints of curves for which the condition of validity for eq. 6′ ($K_A' > K_H$) no longer applies, the equation may be simplified to

$$K'_A = \frac{a_H^2 - K_H' a_H}{K_H'} \qquad (6'')$$

The corresponding simplification of eq. 8′, valid when $a_H << K_H$, (throughout practically the entire range of pH values in which the midpoints of the curves occur) is

$$K_A' = \frac{a_H^2 + K_H' a_H}{K_H'} \qquad (8'')$$

The calculation of K_A', the reciprocal of the affinity, thus depends in either case on an accurate knowledge of K_H', the intrinsic acidity constant of the carboxyl groups of the fiber. By assigning the previously determined approximate value at 0° C. to this constant, the relation of K_A' to the pH of the midpoint has been calculated for this temperature according to both equations. The result of this calculation is plotted logarithmically in Fig. 2, in

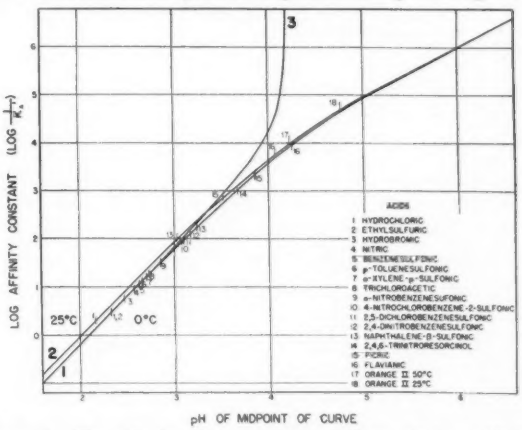


Figure 2—The relation between the position of the acid titration curve with respect to pH and the affinity of the anion of the acid for protein. Curves 1 and 2 represent eq. 8″ with numerical values of K_H' at 0° C. and 25° respectively inserted. Curve 3 represents eq. 6″ with the numerical value of K_H' at 0° C.' Curves 1 and 3 coincide at low pH values.

which the midpoint pH, and therefore the value of the affinity constant, according to both eq. 6″ and eq. 8″ has been indicated for each of the acids represented in Fig. 1.

It is apparent that in the region of low pH values both equations give practically identical results. The curves approach the same straight line having a slope of 2. In this region the ratio of affinities between two anions is given with a high degree of approximation by the antilogarithm of twice the difference in pH between the midpoints of the titration curves obtained with the acids corresponding to the anions.

At pH values above 3, however, the curves diverge appreciably, and at pH values above 4.2 (pK_H' at 0° C.), no physically real values of K_A' are given by eq. 6″. Obviously, however, the conditions under which eq. 6 and 6″ are valid ($K_A' > K_H$) are no longer realized when the

*When salt is present, this equation gives the amount of acid *plus salt* combined since the excess negative charge due to WA^- may be neutralized in part by adsorbed cations other than hydrogen ions. The amount of salt combined is large only when anions of high affinity are present, or when high concentrations are used, so that the titration curve falls in a region of high pH values.

**In all the calculations that follow the reasonable assumption is made that K_H' is an intrinsic property of the protein, and is not appreciably affected by the nature of the anion combined[36], footnote 15.

curves are displaced to such high ranges of pH. Since both equations lead to essentially the same relation between K_A' and midpoint pH in the range of pH in which eq. 6" is nearly exact, all calculations of affinity in this paper have therefore been based on eq. 8", regardless of the range of pH in which the midpoint of a given titration curve may lie.

It should be noticed that as the affinity increases, the difference in the positions of the curves produced by a given increase in affinity becomes larger until ΔpH, which at one extreme is equal to $-\frac{1}{2}\ \Delta pK_A'$ at the other extreme approaches $-\Delta pK_A'$. As the latter limit is approached closely, the affinity constant becomes independent of the value assigned to K_H' and is equal to the midpoint a_H. It will be shown later that the affinities of commercial acid dyes probably fall in this range; thus, the comparison of their affinities relative to one another may be much simplified.

Included in Fig. 2 is a third curve, also representing eq. 8", in which the value 9.32×10^{-5} has been used for K_H' instead of the value 6.30×10^{-5}. This curve shows the relation between midpoint pH and log K_A', at a temperature of 25°[38], and also illustrates the relatively large effect of variations in K_H' on the estimation of K_A' when the latter is large, and the virtual absence of this effect when the latter is small.

By making use of the curve which represents eq. 8" at 0° C., values of K_A' which correspond to each of the acids represented in Fig. 1 have been determined. These values are given in the fifth column of Table 1. The ion with least affinity, chloride, has a value of K_A', 0.35; picrate, the most tightly bound monovalent ion studied at this temperature, has an affinity almost one thousand times as great as that of chloride, and the divalent flavianate ion has an affinity still higher*. It is apparent from the results summarized in Table I that the affinity of the anion tends to rise as the molecular weight increases, but the tendency is not regular and exceptions in the series occur.

Since the experimental curves are broader than those predicted by the much simplified equations, the use of the equations in analyzing the shifts in position of the curves would be rendered more significant, if the progressive change in their slopes could also be shown to be a consequence of the same differences in K_A' which result in their displacements to progressively higher pH values. This demonstration has been made by substituting representative numerical values in eqs. 6 and 8. For the sake of definiteness, the value, 0.01 has been assigned to the ratio $\frac{K_H'}{K_H}$ which has been shown to have a range of reasonable values of 0.01 to 0.001. The result of this calculation with six different values of K_A' is shown in Fig. 3. The calculated curves simulate the curves drawn through the experimental data not only in the relation between the magnitudes of their displacements from one another, but also in the relation of their slopes. Thus, the curves representing low values of K_A' (high affinities) are everywhere less steep than those which correspond to high values of K_A' (low affinities). The progressive change in slope at the midpoints of the experimental curves is in the same direction. The qualitative agreement with experiment of these calculations would not be altered by the assignment of other reasonable numerical values to the ratio $\frac{K_H'}{K_H}$.

(b) *Alternative Method of Calculating Relative Anion Affinities*

In the 6th column of Table 1 is given the ratio of the affinity of the anion of each acid to that of chloride ion.

*For comparison, the affinity of the divalent flavianate ion is expressed in the same units as the affinities of the monovalent acids in the table.

TABLE 1
Calculation of Anion Affinity Constants ($1/K_A'$) at 0° C.

Acid	*Molecular Weight*	*pH of Midpoint*	*— Log K_A' from Eq. 8"*	K_A'	*Affinity Relative to Aff. of Chloride Ion*	*pH at Which Curve Crosses HCl 0.1 Molal Curve*	*Affinity Relative to Aff. of Chloride Ion (Method of Crossing Point)*
Hydrochloric	36.5						
Ethylsulfuric	126.1	2.33	0.45	0.35			
Hydrobromic	80.9	2.47	0.74	0.18	1.9		
Nitric	63.0	2.58	0.96	0.11	3.2	1.71	3.9
Benzenesulfonic	158.2	2.63	1.05	0.089	3.9	1.71	3.9
p-Toluenesulfonic	172.2	2.66	1.10	0.080	4.4	1.73	4.1
o-Xylene-*p*-sulfonic	186.2	2.71	1.21	0.062	5.6	1.83	5.2
Trichloroacetic	163.4	2.73	1.25	0.056	6.2	1.93	6.6
o-Nitrobenzenesulfonic	203.2	2.86	1.50	0.032	11.0	2.05	8.6
4-Nitrochlorobenzene-2-sulfonic	237.6	3.08	1.93	0.0118	29.7	2.68	36.8
2,5-Dichlorobenzenesulfonic	227.1	3.09	1.95	0.0112	31.2	2.68	36.8
2,4-Dinitrobenzenesulfonic	248.2	3.17	2.09	0.0081	43	2.82	51
Naphthalene-β-sulfonic	208.2	3.24	2.23	0.0059	59	3.05	86
2,4,6-Trinitroresorcinol	245.1	3.67	3.02	0.00096	366		
Picric	229.1	3.86	3.34	0.00046	758		
Flavianic	314.2	4.24	†3.94	†0.000115	†3020		

†Calculated, for comparison, as if the anion were monovalent. Since equilibrium was not attained in 23 days, the relative affinity reported is minimal.

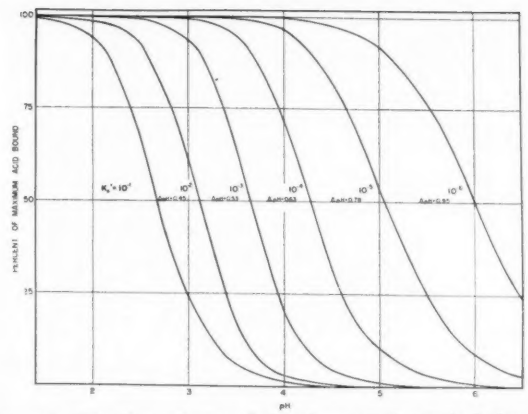


Figure 3—The dependence of the slope of theoretical titration on the protein-anion dissociation constant. The two leftmost curves are calculated from eq. 6 and the others from eq. 8. The change from eq. 6 to eq. 8 between $K_A' = .01$ and $K_A' = .001$ has been made in order to preserve consistency with the assumption $\frac{K_H'}{K_H} = .01$.

This has been tabulated because the same relative affinity factor may be calculated directly from the experimental results represented in Fig. 1 by combining them with the data for hydrochloric acid at constant chloride concentrations in an earlier paper[36]. The values so obtained thus do not depend on the equations just used.

This alternative method of calculation is made possible by the fact that curves obtained in the presence of constant anion concentrations are less steep than those obtained in the absence of salt and therefore cover a wider range of pH values. Thus, for example, a curve representing results obtained with hydrochloric acid at a constant concentration (0.1 *M*) of chloride ions would intersect all of the curves represented in Fig. 1 with the exception of the curve for flavianic acid. The intersections with the curves for hydrobromic acid and picric acid would occur so near the extremes of the 0.1 *M* curve that they are not well suited to the present calculation but all of the other intersections may be used. At the point of intersection of the 0.1 *M* hydrochloric acid curve with any curve in Fig. 1 the same amounts of two different acids are bound by the wool at the same pH. The same degree of combination with two different anions is thus brought about by the presence of different amounts of the respective anions by virtue of the difference of affinity for wool characterizing these anions. It follows from the range of ratios of K_H'/K_H which has been adopted elsewhere that the ratio of the activities of the two anions in the two different experiments is inversely proportional to the ratio of the respective anion affinity constants. The anion affinity ratios which are obtained should be the same as those given in column 6 of Table 1.

The activity of potassium chloride at 0.1 *M* at 0° is 0.0768[18]. The assumption has been made that the activity of the anion in each experiment made in the absence of salt is equal to the activity of the hydrogen ion. It is very unlikely that this assumption is true, but the experimental value, pH, is related to the activities of both ions and methods of obtaining individual ion activities are at present unknown. By dividing 0.0768 by the antilogarithm of the negative of the pH, the values given in the last column of Table 1 have been obtained. In view of the fact that the intersecting curves were drawn freehand and that the two kinds of data were obtained with different lots of wool, the agreement of the values in the last column were those in column 6 is satisfactorily close*

3. Results Obtained in the Presence of Salt

It should be possible to predict the displacements that would be found if the same acids were used in the presence of constant concentrations of their anions, added as neutral salts. By starting with eqs. 1 and 7 instead of eqs. 6 and 8, and making the same simplification which was carried out in arriving at eqs. 6'' and 8'', formulas which give the relation between K_A' and the midpoint pH when the measurements are made in the presence of any constant concentration of anions are obtained:

$$K_A' = a_A \frac{a_H - K_H'}{K_H'} \qquad (1'')$$

$$K_A' = a_A \frac{a_H + K_H'}{K_H'} \qquad (7'')$$

In these equations the quadratic term a_H^2, found in eqs. 6'' and 8'' does not appear. In the range of relatively low affinities, therefore, a given change in K_A' will, in the presence of constant anion concentrations, produce twice the pH change which corresponds to this change in K_A' in the absence of salt. With anions having higher affinities, however, displacements in midpoint pH values obtained in the presence and absence of salt will differ by less than a factor of 2. These predictions have been tested by making measurements with a number of the acids included in Table 1, in the presence of 0.1 *M* concentrations of their anions, added as the potassium salts. The results of these titrations are represented graphically in Fig. 4. As the figure shows clearly, the difference in the pH values of the midpoints of the hydrochloric acid and benzenesulfonic acid curves are twice as great for curves obtained in the presence of salt as for those obtained in its absence. The difference between the midpoints of the hydrochloric acid and naphthalenesulfonic acid curves are somewhat less than twice as great in the presence of salt as in its absence. Both differences thus agree with the predictions of the equations.

Similar experiments have also been made at 25° C. with Orange II in the presence of a constant concentration of its colored anion, added as the sodium salt. The affinity of this anion is so great, that it combines with the protein in considerable amounts at pH values at which

*A third method of calculating relative affinities has also been made use of. Mixtures of acids have been equilibrated with wool, and the relative amounts of the two acids combining have been determined. The relative affinities of the two anions calculated from this ratio agree closely with the results of the methods described in this paper. Since the method has thus far been applied to experiments with acids for which no data are given in the present paper, and since work with these acids is still in progress, no examples of these calculations are given here.

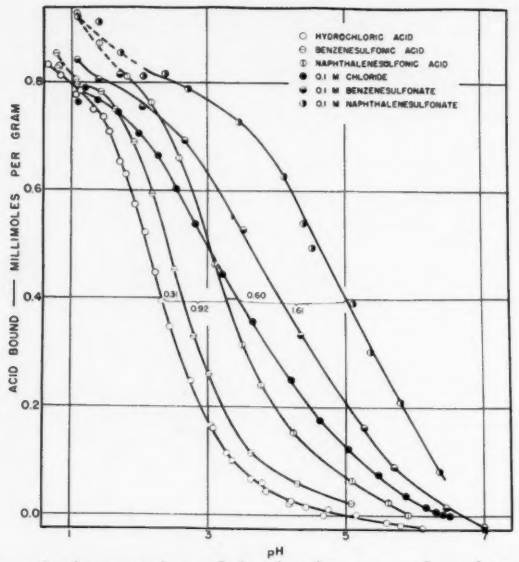


Figure 4—A comparison of the titration curve of wool protein with three different acids in the absence of salt at 0° C., with the curves obtained at the same temperature in the presence of a constant concentration (0.1 *M*) of the anions of the acids.

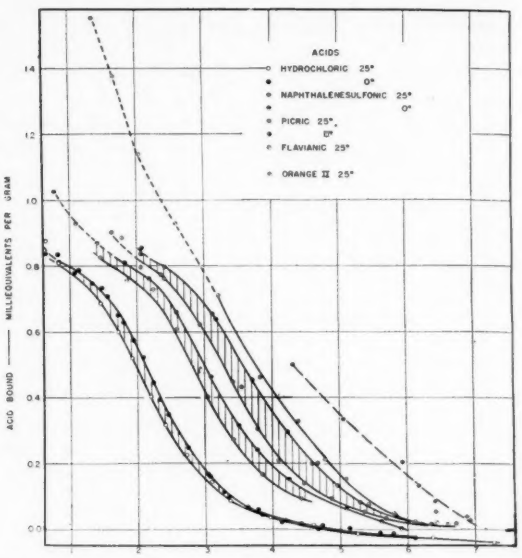


Figure 5—A comparison of the titration curves of wool with various acids at 0° C. and 25° C.

only small amounts of hydrogen ion are normally taken up. The resulting negative charge on the fiber is neutralized, as in all the cases covered by eqs. 7 and 8, by adsorption of positive ions. In the presence of salt, appreciable quantities of sodium ions, as well as hydrogen ions, are bound. Thus, eq. 7 no longer gives the amount of acid taken up alone, but the sum of the amounts of acid and of salt. Since the analysis of these data thus requires the development of a technique for the accurate estimation of this sum, the data obtained with Orange II at a constant concentration of its anion are not included in the present paper. The great steepness of the 0.1 *M* naphthalenesulfonate curve in Fig. 4 is due, in part, to combination with small amounts of salt at high pH values. These should have been added to the amounts of acid combined in order that the resulting curve should be described by eq. 7.

4. Effect of Temperature on Anion Affinity

It has been shown earlier[38], that a comparison of titration data obtained at two different temperatures permits an estimation of heat changes accompanying the dissociation of the protein-anion complex. Measurements at 25° C. have therefore been made with four acids of widely different affinities. The results, represented in Fig. 5 together with the 0° data for three of the acids, also provide a basis of comparison for acids which it is impractical to investigate at 0° C. Results obtained at 25° C. with one such acid, Orange II, are also included in the figure. As the figure makes clear, the effect of temperature on the position with respect to pH of the curves obtained with picric acid (0.34 pH unit) is considerably greater than its effect on the curves for naphthalenesulfonic acid (0.21 unit) which in turn show a greater effect than the curves for hydrochloric acid (0.16 unit). No estimate is attempted of the effect of temperature on the position of curves for flavianic acid, since the data obtained at 0° C. with this acid probably did not represent final equilibrium.

The curve at 25° C. for flavianic acid requires further explanation. When plotted, as in Fig. 5, as *milliequivalents* per gram against pH, the data run nearly parallel to those for picric acid at the same temperature up to values of acid combined of at least 0.65 millimole per g. This furnishes a very strong indication that in this range of pH values the flavianate ions combining with the wool are predominantly doubly ionized, and effectively neutralize two of the positively charged basic groups in the protein. At lower pH values where the data are represented by a broken line, the proportion of the total flavianic acid which is doubly dissociated is so small that a part of the ions which combine with the fiber are monovalent. In the most concentrated solutions combination with the fiber takes place almost entirely with the monovalent ion, so that a plateau at 0.82 *millimole* rather than at 0.82 *milliequivalent* would presumably be found. Since the concentration of flavianic acid is extremely low in the range of pH in which all the ions are predominantly dibasic, it is clear that the affinity of the doubly charged anion is considerably higher than that of the monovalent ion. Trinitroresorcinol also combined with wool as a dibasic acid, when present in greater dilution. To avoid complicating Fig. 1 only the measurements representing the monobasic function of this acid were included in the figure. Similar effects appear to characterize sulfuric acid[34].

The affinities at 25° C. of the acids represented in Fig. 5, calculated from eq. 8'' are given in the first part of Table 2. The values of K_A' listed in column 4 were obtained graphically from the curve for 25° in Fig. 2. Thus the effect of temperature on K_H' has been taken into account. Owing to the inclusion of data for Orange II, the differences of affinity comprised in this table cover almost

TABLE 2
Calculation of Anion Affinity Constants and Heats of Dissociation at 25° and 50° C.

Acid	*pH of Midpoint*	*— Log K_A' from Eq. 8''* 25°	K_A'	*Affinity Relative to Aff. of Chloride*	$\left(\frac{K_A'}{K_A'}\right)_{0°}^{25°}$	$\Delta H_{0°}^{25°}$ *for Anion Dissociation Calories*
Hydrochloric	2.16	0.31	0.49		1.40	2170
Naphthalene-β-sulfonic	3.03	1.97	.0107	46	1.81	3850
Picric	3.52	2.89	.00129	380	2.80	6670
Flavianic	4.07	**3.77	.000170	2880		
*Orange II	4.76	4.68	†.000021	†23400		
		50°			$\left(\frac{K_A'}{K_A}\right)_{25°}^{50°}$	$\Delta H_{25°}^{50°}$
Hydrochloric	2.21	0.38	0.42		0.85	—1,630
*Orange II	4.21	3.97	.000107	3,900	†5.09	†12,500

*Molecular weight 328.3.

**Expressed for purposes of comparison as if the anion were monovalent. Refer to text.

†The Orange II data at 25° C. do not represent equilibrium values. Thus the relative affinity given for this temperature, and the value of $\Delta H_{25°}^{50°}$ are minimal.

eight times as wide a range as those listed in Table 1.

The sixth column of the table gives the ratio of the protein-anion dissociation constants at 25° and 0°. From this ratio, and the van't Hoff equation, the average heat of dissociation in this range of temperature has been calculated. The values so obtained are given in the last column. The heats of dissociation rise from some 2,000 calories in the case of chloride ion to nearly 7,000 calories in the case of picrate. As should be expected more energy is required to dissociate the more tightly bound ions*.

In order to evaluate the effect of temperature on the affinity of Orange II, the amounts of this acid combined by wool were also measured at 50° C, after allowing a period of 5 days for the attainment of equilibrium. The results, together with data for hydrochloric acid at the same temperature are represented graphically in Fig. 6. The maximum amounts combined are in fair agreement with those reported for high concentrations of this dye by Ender and Müller[12]. Included in the figure for the purpose of comparison are the data obtained with Orange II at 25° C. The measurements represented by crosses were obtained at 50° C. by determining the increase in dry weight of the fibers as the result of combination with dye. The small discrepancies between the two sets of results (larger the more acid the solution) are accounted for by the fact that small amounts of amide nitrogen are split off, as a result of the prolonged exposure to dye at this temperature, and affect the apparent dye-uptake estimated from the difference in the acid titer of the dye bath brought about by the wool. The amounts of ammonia produced by comparable exposure to the same concentrations of hydrochloric acid are considerably smaller.

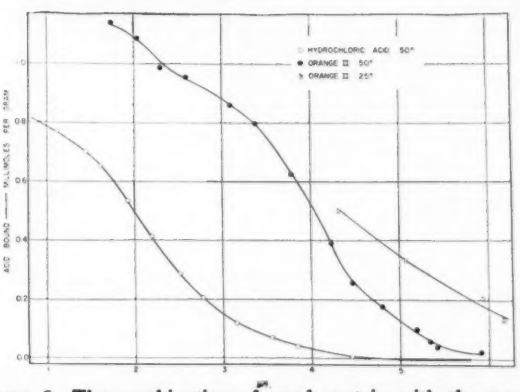


Figure 6—The combination of wool protein with the azo-dye acid Orange II, and with hydrochloric acid as a function of pH at 50° C. The significance of the crosses is explained in the text. For comparison, data obtained with Orange II at 25° C. are included.

A comparison of the titration curve obtained with Orange II at 50° C. with the portion of the curve obtained at 25° C. strongly suggests that the data for the lower temperature do not represent a final equilibrium state. This was clearly the case when higher concentrations of dye were used at 25° C; combination of dye in 35 days at this temperature in no case exceeded 0.63 millimole, even when solutions more acid than those used at 50° C, were employed. The affinity of the dye anion at 25° (listed in the first part of table 2) must therefore be regarded as minimal. Thus, the heat of dissociation of the dye anions is even larger than the value in the table (12,500 calories) and exceeds by a large factor the heats of dissociation of the other ions listed. This large value also suggests an explanation of why the *rates* of dyeing and of stripping are so much affected by temperature. Since the anions are held much more loosely at elevated temperatures (the factor is greater than 5 in the interval 25°C.-50°C.), they are more readily passed on from one combining group in the fiber to another, toward the innermost parts of the fiber from the peripheral portions

*The value of K_H' at 25° was chosen on the basis of the earlier analysis of the effect of temperature on the combination of wool with hydrochloric acid[28], in which it was assumed that the heat of transfer of the ions from the solution to the fiber could be neglected. The heats of dissociation given in the table may therefore be subject to a small correction. Since this correction would be practically invariant, its application would not affect the relative order of the heats of dissociation, nor the great differences between them.

which are first dyed, or in the stripping process in the opposite direction.

(To be concluded)

Wetting Agents—

(Concluded from page 249)

2. It has been shown that some commercial wetting agents tend to make wool felt rapidly in acid solution. Such compounds might be useful as felting aids in acid solution.

3. It has been shown that when the wetting and felting powers of anionic-active wetting agents are compared, good wetting agents will be found to be good felting agents, and vice versa. It is probable that this statement is true only when the wetting and felting tests are run in acid solution and are similar to those described in this paper.

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Technical Notes—

(Concluded from page 262)

wool fiber, and has definitely improved resistance to abrasion. The gain is at least 25 per cent and usually as high as 100 per cent, together with an increase of about 10 per cent in wet and dry strength.

Cross sections under the microscope show a closer packing of the filaments of the treated material, with occasional adherence, and a general smoothing and rounding of the filaments. The closer approach of the filaments to each other is a great factor in the change of feel but the goods seem definitely fuller without the addition of any foreign material. This would seem to mean a residual swelling in the fibers, probably at the expense of some shrinkage but it is not explained in this way.

The Adry treated goods are easily dyed and a saving of 20-30 per cent of dye is claimed. The process can be followed by other finishing treatments to add other effects. The results of various tests are given to show that there really is a substantial gain in resistance to abrasion and that it means an actual gain in durability. Gains in different kinds of goods ran from 54 to 188 per cent in resistance to abrasion, above the original value. Washing ten times before the test was made caused a decreased resistance in some cases but was followed by greater resistance in others. In all cases the washed samples were far more resistant than the goods without the special treatment when similarly washed.

• CLASSIFIED • ADVERTISEMENTS

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HELP WANTED: Textile Dyer wanted for textile mill in Allentown, Pa. Must be graduate chemical engineer and have ability to handle highly diversified types of dyeing. Write giving full details, age, education, experience, salary expected, etc. Box No. 311, American Dyestuff Reporter, 440 Fourth Ave., New York, N. Y.

POSITION WANTED: Dyer, 15 years' experience with two of the countries leading organizations. Head dyer for the past 10 years. Experience on all types of yarns, rayon, wool, silk, cotton and mixed yarns. Well versed in the application of dyestuffs to these different textile fibers: Direct, Sulfur, Napthols, Chrome, Union and Vats. Also overseer on Mercerizing, Bleaching and Finishing. Age 34, married. Will go anywhere. References. Box No. 312, American Dyestuff Reporter, 440 Fourth Ave., New York, N. Y.

Young man, 34 years of age and married, would like to obtain position as foreman or assistant to dyer. Have had six years' practical experience in Dyehouse on K. & W. machines, hand kettles and Lovzons. Education, three years' course in Chemistry and Dyeing at Philadelphia Textile School. Write Box No. 313, American Dyestuff Reporter, 440 Fourth Ave., New York, N. Y.

POSITION WANTED: Energetic textile school graduate, three years' experience assistant dyer and chemist. Sincerely seeking opening where hard work and reliability may lead to future. Box No. 314, American Dyestuff Reporter, 440 Fourth Ave., New York, N. Y.

PLANT SUPERINTENDENT WANTED: Must be thoroughly familiar with all phases of a dyeing and finishing plant which handles all types of Acetate fabric and Rayon fabric, both on box and jigg. Must be thoroughly competent. Write Box No. 315, American Dyestuff Reporter, 440 Fourth Ave., New York, N. Y.